AMERICAN SOCIETY
for
TESTING MATERIALS

(Reg. U.S. Pat. Office)

PROCEEDINGS

VOLUME 59
1959

COMMITTEE REPORTS
TECHNICAL PAPERS

Published by the
AMERICAN SOCIETY FOR TESTING MATERIALS
1916 Race St., Philadelphia 3, Pa.

Engin. Library
TA
401
.A51
v.59

© BY AMERICAN SOCIETY FOR TESTING MATERIALS 1960

Library of Congress Catalog Card Number: 8-5854

Printed in Baltimore, U. S. A.
April, 1960

FOREWORD

The *Proceedings* of the American Society for Testing Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings,* together with discussion.

The table of contents and subject and author indexes cover all papers and reports published by the Society during the current year, which in addition to those appearing in the *Proceedings* include those accepted for publication in the ASTM BULLETIN or in Special Technical Publications.

A list of the Special Technical Publications published by the Society in 1959 is given on page 1413 of this volume. This supplements the lists appearing in the *Proceedings* from 1948 to 1958 covering all special publications published by the Society up to and including 1958.

Nothing contained in any publication of the American Society for Testing Materials is to be construed as granting any right, by implication or otherwise, for manufacture, sale, or use in connection with any method, apparatus, or product covered by Letters Patent, nor as insuring anyone against liability for infringement of Letters Patent.

The Society is not responsible, as a body, for the statements and opinions advanced in this publication

CONTENTS

PROCEEDINGS

PAGE

Summary of Proceedings of the Sixty-second Annual Meeting 1

Summary of Proceedings of the Third Pacific Area National Meeting 36

Summary of Proceedings of Pittsburgh Spring Meeting—Symposium on Testing of Window Assemblies (*ASTM STP No. 251* 1337

Annual Address by the President—K. B. Woods 56

Annual Report of the Board of Directors 61

- Appendix I. Report of the Auditors for the Fiscal Year November 27, 1957 to November 28, 1958 81
- Appendix II. Report of Administrative Committee on Standards 95
- Appendix III. Report of Administrative Committee on Papers and Publications. 102
- Appendix IV. Report of Administrative Committee on District Activities 106
- Appendix V. Report of Administrative Committee on Research 107
- Appendix VI. Report of Administrative Committee on Simulated Service Testing 109

COMMITTEE REPORTS

Ferrous Metals

Report of Committee A-1 on Steel 110

- Appendix. Proposed Recommendations Affecting Standards on Steel 116

Report of Committee A-3 on Cast Iron 124

Report of Committee A-5 on Corrosion of Iron and Steel 127

- Appendix. Preliminary Data on 1958 Atmospheric Exposure of Hardware Specimens 133

Report of Committee A-6 on Magnetic Properties 156

Report of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys 158

Non-Ferrous Metals

Report of Committee B-1 on Wires for Electrical Conductors 163

Report of Committee B-2 on Non-Ferrous Metals and Alloys 171

Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys 174

- Appendix I. Report of Subcommittee VI on Atmospheric Corrosion (1957 Test Program) 176
- Appendix II. Report of Subcommittee VII on Corrosiveness of Various Atmospheric Test Sites as Measured by Specimens of Steel and Zinc 183
- *Second Progress Report: Measurement of Surface Moisture—P. J. Sereda* (*See* ASTM BULLETIN, *No. 238, May, 1959, p. 61*)

Report of Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance and Electrical Contacts 202

Report of Committee B-5 on Copper and Copper Alloys, Cast and Wrought 205

- Appendix I. Recommendations Affecting Standards on Copper and Copper Alloys, Cast and Wrought 209
- Appendix II. Effect of Gage on Rockwell Hardness Testing of Brass Strip—Delmar E. Trout 212
- Appendix III. Magnetic Properties of Copper Alloys—F. G. Parker, R. M. Winter, and J. A. LaPorta 230

Report of Committee B-6 on Die-Cast Metals and Alloys 262

Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought 263

- Appendix I. Proposed Recommendations Affecting Standards for Light Metals and Alloys, Cast and Wrought 267
- Appendix II. Chromic Acid Anodizing Characteristics of Wrought Aluminum Alloys—George E. Best, J. G. Hecker, Jr., John W. McGrew, and R. V. VandenBerg 277

PAGE
Appendix III. Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings—L. H. Adam and Marie Dougherty 289
Report of Committee B-8 on Electrodeposited Metallic Coatings 315
Appendix. Standardizing the Preparation of Electrodeposits on Test Panels for Corrosion Testing—A. K. Graham and H. L. Pinkerton 317
Report of Committee B-9 on Metal Powders and Metal Powder Products 330

Report of Advisory Committee on Corrosion 333
Report of Joint Committee on Effect of Temperature on the Properties of Metals 339

Cementitious, Ceramic, Concrete, and Masonry Materials

Report of Committee C-1 on Cement 345
Appendix I. Short-Time Tests of Mortars for Controlling SO_3 in Portland Cement at Optimum Value 349
Appendix II. The Cement Reference Laboratory (1929–1959)—J. R. Dise 369
Report of Committee C-3 on Chemical-Resistant Mortars 378
Report of Committee C-4 on Clay Pipe 380
Report of Committee C-7 on Lime 381
Report of Committee C-8 on Refractories 383
Report of Committee C-9 on Concrete and Concrete Aggregates 387
Proposed Tentative Method of Test for Cement Content of Freshly Mixed Concrete (*See* ASTM BULLETIN, *No. 239, July, 1959, p. 48*)
Report of Committee C-11 on Gypsum 393
Report of Committee C-12 on Mortars for Unit Masonry 395
Report of Committee C-13 on Concrete Pipe 396
Appendix. Recommendations Affecting Standards on Concrete Pipe 398
Report of Committee C-15 on Manufactured Masonry Units 402
Report of Committee C-16 on Thermal Insulating Materials 406
Report of Committee C-17 on Asbestos-Cement Products 410
Report of Committee C-19 on Structural Sandwich Constructions 412
Report of Committee C-20 on Acoustical Materials 414
Report of Committee C-21 on Ceramic Whitewares and Related Products 416
Report of Committee C-22 on Porcelain Enamel 418
Proposed Specification for Sealing Compound for the Building Trade (*Polysulfide Base or Equivalent*) (See ASTM BULLETIN, *No. 242, Dec. 1959, p. 46*)

Miscellaneous Materials

Report of Committee D-1 on Paint, Varnish, Lacquer, and Related Products 420
Report of Committee D-2 on Petroleum Products and Lubricants 431
Separation of Oil from Lubricating Greases in Storage—A Report of Section IV of Technical Committee G (*See* ASTM BULLETIN, *No. 233, October 1958, p. 27*)
Report of Committee D-3 on Gaseous Fuels 447
Report of Committee D-4 on Road and Paving Materials 449
Report of Committee D-5 on Coal and Coke 454
Report of Committee D-6 on Paper and Paper Products 457
Report of Committee D-7 on Wood 460
Report of Committee D-8 on Bituminous Waterproofing and Roofing Materials 466
Report of Committee D-9 on Electrical Insulating Materials 468
Report of Committee D-10 on Shipping Containers 477
Report of Committee D-11 on Rubber and Rubber-Like Materials 479
Melting Range of Rubber Chemicals—Prepared by Task Group on the Analysis of Rubber Chemicals (*See* ASTM BULLETIN, *No. 231, July 1958, p. 92*)
Tension Testing of Rubber Rings—F. L. Roth and R. D. Stiehler (*See* ASTM BULLETIN, *No. 233, October 1958, p. 38*)
Report of Committee D-12 on Soaps and Other Detergents 489
Report of Committee D-13 on Textile Materials 492
Appendix. Recommendations Affecting Standards on Textile Materials 494
Report of Committee D-14 on Adhesives 500
Report of Committee D-15 on Engine Antifreezes 503
Report of Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials 505
Appendix I. Cooperative Data on Method for Water in Phenol 509

PAGE

Appendix II. Proposed Method of Test for *Ortho*-Xylene, *Meta*-Xylene, *Para*-Xylene, and Ethylbenzene in Nitration Grade Xylenes by Infrared Spectrophotometry 510
Appendix III. Results on Thiophene in Benzene Cooperative Program Using Proposed Method 514
Report of Committee D-17 on Naval Stores 515
Report of Committee D-18 on Soils for Engineering Purposes 518
Report of Committee D-19 on Industrial Water 523
Report of Committee D-20 on Plastics 532
Report of Committee D-21 on Wax Polishes and Related Materials 542
Report of Committee D-23 on Methods of Atmospheric Sampling and Analysis 544
Report of Committee D-24 on Carbon Black 546
Report of Committee D-25 on Casein and Similar Protein Materials 548
Report of Committee D-26 on Halogenated Organic Solvents 549
Report of Committee D-27 on Electrical Insulating Liquids and Gases 550
Report of ALCA-ASTM Joint Committee on Leather 555

Miscellaneous Subjects

Report of Committee E-1 on Methods of Testing 557
Report of Committee E-2 on Emission Spectroscopy 569
Appendix I. Suggested Method for Spectrochemical Analysis of Antimony by the Point-to-Plane Spark Technique 573
Appendix II. Suggested Method for Spectrochemical Analysis of Magnesium-Base Alloys by the Point-to-Plane Spark Technique Using a Grating Spectrograph 577
Report of Committee E-3 on Chemical Analysis of Metals 581
Report of Committee E-4 on Metallography 586
Report of Committee E-5 on Fire Tests of Materials and Construction 588
Report of Committee E-6 on Methods of Testing Building Constructions 591
Report of Committee E-7 on Nondestructive Testing 593
Report of Committee E-9 on Fatigue 595
Appendix. Proposed Revision of Section II, Manual on Fatigue Testing, *STP 91*. 596
Recent Survey of Current Activities on Fatigue (*See* ASTM BULLETIN, *No. 235, January 1959, p. 26*)
Report of Committee E-11 on Quality Control of Materials 605
Report of Committee E-12 on Appearance 606
Report of Committee E-13 on Absorption Spectroscopy 607
Appendix I. Proposed Recommended Practice for General Techniques of Infrared Quantitative Analysis 610
Appendix II. Proposed Recommended Practice for General Techniques of Ultraviolet Quantitative Analysis 622

Materials for Specific Applications

Report of Committee F-1 on Materials for Electron Tubes and Semiconductor Devices 628

TECHNICAL PAPERS

Edgar Marburg Lecture

New Polymers—New Problems—H. F. Mark (*intended for separate publication only*)

Gillett Memorial Lecture

The Role of Dislocations in Plastic Deformation—J. C. Fisher (*issued only as separate pamphlet*)

Fatigue

Tension, Compression, and Fatigue Properties of Several Steels for Aircraft Bearing Applications—G. Sachs, R. Sell, and W. F. Brown, Jr. 635
Discussion 658
The Effect of Stress Cycling on the Static Mechanical Properties of SAE 4340 Steel—J. Marin, P. Borachio, and U. A. Rimrott 662
Discussion 673

PAGE

Some Observations on the Plastic Work Required to Fracture Stainless Steel Under Cyclic Loading—D. E. Martin and J. Brinn 677
Discussion 685
Techniques and Equipment for Fatigue Testing at Very High Frequencies—E. A. Neppiras 691
Discussion 709
A Method for Determining the Fatigue Limit of Metals by Means of Stepwise Load Increase Test—N. Enomoto 711
Discussion 721
Effect of Internal Heating on the Fatigue Life of Titanium—J. P. Romualdi and E. D'Appolonia 723
Discussion 731
Crack Propagation in Cold-Rolled Aluminum Sheet—H. A. Lipsitt, F. W. Forbes, and R. B. Baird 734
Discussion 748
Tensile and Fatigue Properties of Laminate Sheet Structures—R. B. Baird, F. W. Forbes, and H. A. Lipsitt 755
Fatigue Tests on Three Cast Irons at Elevated Temperatures—R. P. Felgar 767

Metals

AISI Type 304L Stainless Steel with Improved Strength—R. R. Brady 774
Discussion 785
Relationships Between Composition and Properties of Austenitic Chromium-Manganese-Carbon-Nitrogen Stainless Steels—A. Kasak, C. M. Hsiao, and E. J. Dulis 786
Discussion 802
Property Relationships of Some Cast and Forged Chromium-Manganese-Nickel-Nitrogen Steels Containing 18 per cent Chromium—K. Haefner, A. F. Lahr, W. L. Meinhart, and J. J. Kanter 804
Discussion 815
Effect of Cold Rolling and Stress Relief on the Sharp Edge Notch Tensile Characteristics of Austenitic Stainless Steel Sheet Alloys—G. B. Espey, A. J. Repko, and W. F. Brown, Jr. 816
Discussion 833
The Sharp Edge Notch Tensile Strength of Several High-Strength Steel Sheet Alloys—G. B. Espey, M. H. Jones, and W. F. Brown, Jr. 837
Discussion 872
Experimental Determination of Energy Release Rate for Notch Bending and Notch Tension—J. D. Lubahn 885
Discussion 914
Effect of Composition and Processing Variables on Some Mechanical Properties of Large Nickel-Molybdenum-Vanadium Rotor Forgings—K. H. Kramer and J. E. Steiner 916
Behavior of Ductile Cast Iron Under Compressive Stress—Paul K. Trojan, R. A. Flinn, and D. J. Reese 922
The Annealing Behavior of 70-30 Brass Strip (Cartridge Brass, 70 per cent)—Verne Pulsifer 930
The Creep and Rupture Properties of Three Cast Copper-Base Alloys Between 550 and 650 F (290 and 345 C)—H. V. Kinsey 940
Effect of Gage on the Rockwell Hardness Testing of Brass Strip—D. E. Trout (Appended to B-5 Report, p. 212)
Magnetic Properties of Copper Alloys—F. G. Parker, R. M. Winter, and J. A. La-Porta (Appended to B-5 Report, p. 230)
Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys—L. H. Adam and M. Dougherty (Appended to B-7 Report, p. 289)
Density Measurements on Strain-Cycled 2S Aluminum—J. F. Tavernelli and L. F. Coffin, Jr. 952
Chromic Acid Anodizing Characteristics of Wrought Aluminum Alloys—G. E. Best, J. G. Hecker, R. V. Vandenberg, and J. W. McGrew (appended to the B-7 Report, p. 277)
Standardizing the Preparation of Electrodeposits on Test Panels for Corrosion Testing—A. K. Graham and H. L. Pinkerton (appended to the B-8 Report, p. 317)
Marine Atmosphere Exposure of Galvanic Couples Involving Magnesium—A. Gallaccio and I. Cornet (ASTM Special Technical Publication No. 255)

PAGE

Literature Surveys on Influence of Stress Concentrations and Elevated Temperatures and the Effects on Non-Steady Load and Temperature Conditions on the Creep of Metals (ASTM Special Technical Publication No. 260)

Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels (ASTM Special Technical Publication No. 264)

Papers on Effect of Temperature

Creep-Rupture Behavior of Notched and Unnotched Specimens of Types 304, 316, and 321 Austenitic Stainless Steels—F. Garofalo 957

Effect of Environment on Creep and Creep-Rupture Behavior of Several Steels at Temperatures of 1000 to 1200 F—F. Garofalo 973

Discussion 983

Effect of Notch Geometry on Tensile Properties of Annealed Titanium at 100, 25, −78, and −196 C—G. W. Geil and N. L. Carwile 985

Discussion 1013

Stress-Strain Properties of Selected Titanium Metals—E. P. Klier and C. P. Gazzara 1015

Discussion 1029

Properties of 70-30 Copper-Nickel Alloy at Temperatures Ranging up to 1050 F—W. F. Simmons, B. J. Sirois, D. N. Williams, and R. I. Jaffee 1035

Discussion 1046

Mechanical Properties of a High-Purity Lead and a 0.058 per cent Copper-Lead Alloy at Elevated Temperatures—T. E. Tietz 1052

Discussion 1071

Symposium on Electron Metallography

Summary of Proceedings of the Symposium on Electron Metallography (*ASTM Special Technical Publication No. 262*) 1072

Symposium on Electroless Nickel Plating

Summary of Proceedings of the Symposium on Electroless Nickel Plating (*ASTM Special Technical Publication No. 265*) 1073

Symposium on Microscopy

Summary of Proceedings of the Symposium on Microscopy (*ASTM Special Technical Publication No. 257*) 1074

Cement and Concrete

An Approach to the Problem of Evaluation of Plasticity and Workability of Pastes and Mortars—A. D. Conrow 1075

Discussion 1086

Quantitative Determination of the Four Major Phases in Portland Cements by X-ray Analysis—Stephen Brunauer, L. E. Copeland, D. L. Kantro, C. H. Weise, and Edith G. Schulz 1091

Short Time Tests of Mortars for Controlling SO_3 in Portland Cement at Optimum Value (appended to C-1 Report, p. 349)

Control of Gypsum in Portland Cement—Bailey Tremper 1101

Discussion 1107

Acetate Processed Portland Cement—B. E. Kester 1111

Discussion 1118

Statistical Aspects of the Cement Testing Program—W. J. Youden 1120

Statistical Evaluation of Interlaboratory Cement Tests—J. R. Crandall and R. L. Blaine 1129

Cement Reference Laboratory (1929–1959)—J. R. Dise (Appended to C-1 Report, p. 369)

Fundamental Characteristics of Pulverized Coal Fly Ashes—L. John Minnick 1155

Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction—Leonard Pepper and Bryant Mather 1178

Discussion 1202

Studies of Limestone Aggregates by Fluid-Flow Methods—W. L. Dolch 1204

Discussion 1214

Effect of Length to Diameter Ratio on Compressive Strength—An ASTM Cooperative Investigation—Clyde E. Kesler 1216

PAGE
Discussion 1229
Stresses in Deep Beams Subjected to Central and Third-Point Loading—M. E. Raville and F. J. McCormick 1230

General Testing

Development of ASTM Standard Reference Blocks for Ultrasonic Inspection—F. C. Panian and H. E. Van Valkenburg 1237
Discussion 1259
A Cooperative Study of the Hardness Testing of Cemented Carbides—Betty M. Caugherty, H. T. Oatman, and O. W. Reen 1261
Discussion 1275
Experimental Observations on the Relation Between Polarization Resistance and Corrosion Rate—Milton Stern and Edward D. Weisert 1280
Discussion 1291
The Tensile Properties of Some Engineering Materials at High Rates of Strain—Arthur L. Austin and Robert F. Steidel, Jr. 1292
Discussion 1307
Compression Testing at Constant True Strain Rates—J. E. Hockett 1309
Discussion 1317
Compression Tests of Wood at Elevated Temperatures—S. H. Ingberg 1320

Symposium on Education in Materials

Summary of Proceedings of the Symposium on Education in Materials (*ASTM Special Technical Publication No. 263*) 1330

Symposium on New Visual Aids for Standardizing and Communicating Product Appearance

Summary of Proceedings of the Symposium on New Visual Aids for Standardizing and Communicating Product Appearance (*ASTM Special Technical Publication No. 258*) 1331

Bituminous Paving Materials

Summary of Proceedings of Bituminous Paving Materials (Symposiums on Bituminous Binders, Paving Mixtures, Road and Paving Papers) (*ASTM Special Technical Publication No. 252*) 1332

Soils—1959

Summary of Proceedings of Soils (Symposiums on Time Rates of Loading in Soil Testing, Atterberg Limits, and Sessions on Soils (Sixty-second Annual Meeting and Third Pacific Area National Meeting) (*ASTM Special Technical Publication No. 254*) 1334

Symposium on Identification of Water-Formed Deposits

Summary of Proceedings of the Symposium on Identification of Water-Formed Deposits (*ASTM Special Technical Publication No. 256*) 1336

Symposium on Testing Window Assemblies

Summary of Proceedings of Symposium on Testing Window Assemblies (*ASTM Special Technical Publication No. 251*) 1337

Symposium on Spectroscopic Excitation

Summary of Proceedings of Symposium on Spectroscopic Excitation (*ASTM Special Technical Publication No. 259*) 1338

Symposium on Radioisotopes in Metals Analysis

Summary of Proceedings of Symposium on Radioisotopes in Metals Analysis (*ASTM Special Technical Publication No. 261*) 1339

Symposium on High Voltage Cable Insulation

Summary of Proceedings of Symposium on High Voltage Cable Insulation (*ASTM Special Technical Publication No. 253*) 1340

List of Technical Papers Published in ASTM BULLETIN 1341
Author Index 1345
Subject Index 1376
List of Special Technical Publications 1413

SUMMARY OF PROCEEDINGS

OF THE

SIXTY-SECOND ANNUAL MEETING

ATLANTIC CITY, N. J., JUNE 21–26, 1959

This summary of the Sixty-second Annual Meeting of the American Society for Testing Materials, held at the Chalfonte-Haddon Hall Hotel, Atlantic City, N. J., June 21–26, 1959, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. In all, 36 technical sessions were held.

The registered attendance of the meeting is as follows: Members present or represented, 1708; committee members, 1054; guests, 371; total, 3133; ladies, 391.

The Proceedings are set forth session by session. There were 71 reports and 106 formal papers presented. The record with respect to each has been briefed; the recommendations in the reports have been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives, and proposed revisions of existing standards, and, as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported "adopted as standard" or "adopted as standard, revisions in," it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society.[1] The various recommendations so recorded are included in the Society letter ballot. The actions designated as "accepted as tentative" or "accepted as tentative, revisions in" are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative. Designations that have since been assigned to new tentatives are included as information.

All papers, unless otherwise noted, are published in the *Proceedings*.

While all the items on the program are recorded under the particular session in which they are presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all reports together with the page reference where the actions thereon are recorded. There are also listed the various symposia and special sessions.

---

[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, will be canvassed on September 10, 1959. See Editor's Note, p. 35.

LOCATION OF ACTION ON COMMITTEE REPORTS

PAGE

Committee:
- Board of Directors 8
- A-1 on Steel 17
- A-3 on Cast Iron 18
- A-5 on Corrosion of Iron and Steel 18
- A-6 on Magnetic Properties 10
- A-7 on Malleable Iron Castings 10
- A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys 10

- B-1 on Wires for Electrical Conductors 5
- B-2 on Non-Ferrous Metals and Alloys 18
- B-3 on Corrosion of Non-Ferrous Metals and Alloys 5
- B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts 30
- B-5 on Copper and Copper Alloys, Cast and Wrought 10
- B-6 on Die-Cast Metals and Alloys 11
- B-7 on Light Metals and Alloys, Cast and Wrought 26
- B-8 on Electrodeposited Metallic Coatings 5
- B-9 on Metal Powders and Metal Powder Products 5

- C-1 on Cement 30
- C-3 on Chemical-Resistant Mortars 11
- C-4 on Clay Pipe 11
- C-7 on Lime 19
- C-8 on Refractories 11
- C-9 on Concrete and Concrete Aggregates 19
- C-11 on Gypsum 30
- C-12 on Mortars for Unit Masonry 19
- C-13 on Concrete Pipe 14
- C-15 on Manufactured and Masonry Units 30
- C-16 on Thermal Insulating Materials 14
- C-17 on Asbestos-Cement Products 26
- C-19 on Structural Sandwich Construction 15
- C-20 on Acoustical Materials 15
- C-21 on Ceramic Whiteware and Related Products 15
- C-22 on Porcelain Enamel 15

- D-1 on Paint, Varnish, Lacquer, and Related Products 20
- D-2 on Petroleum Products and Lubricants 31
- D-3 on Gaseous Fuels 20
- D-4 on Road and Paving Materials 32
- D-5 on Coal and Coke 26
- D-6 on Paper and Paper Products 33
- D-7 on Wood 23
- D-8 on Bituminous Waterproofing and Roofing Materials 20
- D-9 on Electrical Insulating Materials 33
- D-10 on Shipping Containers 23
- D-11 on Rubber and Rubber-like Materials 34
- D-12 on Soaps and Other Detergents 23
- D-13 on Textile Materials 23
- D-14 on Adhesives 24
- D-15 on Engine Antifreezes 24
- D-16 on Industrial Aromatic Hydrocarbons and Related Materials 35
- D-17 on Naval Stores 26
- D-18 on Soils for Engineering Purposes 21
- D-19 on Industrial Water 28
- D-20 on Plastics 26

PAGE

Committee:
- D-21 on Wax Polishes and Related Materials........ 24
- D-23 on Cellulose and Cellulose Derivatives........ 24
- D-24 on Carbon Black........ 27
- D-25 on Casein and Similar Protein Materials........ 21
- D-26 on Halogenated Organic Solvents........ 24
- D-27 on Electrical Insulating Liquids and Gases........ 21

- E-1 on Methods of Testing........ 27
- E-2 on Emission Spectroscopy........ 31
- E-3 on Chemical Analysis of Metals........ 18
- E-4 on Metallography........ 19
- E-5 on Fire Tests of Materials and Construction........ 15
- E-6 on Methods of Testing Building Constructions........ 15
- E-7 on Nondestructive Testing........ 28
- E-11 on Quality Control of Materials........ 35
- E-12 on Appearance........ 28
- E-13 on Absorption Spectroscopy........ 31

- F-1 on Materials for Electron Tubes and Semiconductor Devices........ 5

- Joint Committee on Effect of Temperature on the Properties of Metals........ 12
- Joint Committee on Leather........ 28

FIRST SESSION—OPENING SESSION

MONDAY, JUNE 22, 9:30 A.M.

FORMAL OPENING OF THE SIXTY-SECOND ANNUAL MEETING, PRESIDENT K. B. WOODS

The session was formally opened by President K. B. Woods, who called attention to the very complete program of events that were to follow during the ensuing week.

SYMPOSIUM ON EDUCATION IN MATERIALS[2]

Jointly sponsored by the American Society for Engineering Education (ASEE) and ASTM

SESSION CHAIRMAN: K. B. WOODS, PRESIDENT, ASTM

Introduction—K. B. Woods, presented by the author.

The ASTM Viewpoint on Education in Materials—F. L. LaQue, presented by the author.

The Educators' Viewpoint—W. T. Alexander, presented by the author.

Industry's Viewpoint—M. F. Wood, presented by the author.

G. B. Warren, presented by the author.

(*Continued in the Second Session*)

EDUCATION IN MATERIALS LUNCHEON

MONDAY, JUNE 22, 12:00 NOON

TOASTMASTER: PAST-PRESIDENT R. T. KROPF

The guest speaker was Eric A. Walker, President, Pennsylvania State University, whose subject was "Education in Engineering."[2]

---

[2] Issued as separate publication *ASTM STP No. 263.*

SECOND SESSION—SYMPOSIUM ON EDUCATION IN MATERIALS[2]

(Symposium continued from the First Session)

MONDAY, JUNE 22, 2:00 P.M.

SESSION CHAIRMAN: W. T. ALEXANDER, PRESIDENT ASEE (1958–1959)

Nature and Properties of Materials (ASEE Follow-Up Report on Evaluation of Engineering Education)—Glenn Murphy, presented by the author.

Solid State Physics in Relation to Materials Science, Education and Industry—J. E. Goldman, presented by the author.

The Viewpoint of the Civil Engineer—H. A. Lepper, Jr., presented by the author.

Summary of Survey on Projected Degree in Materials Engineering and Related Topics—M. E. Shank, presented by the author.

THIRD SESSION—CONCRETE

MONDAY, JUNE 22, 2:30 P.M.

SESSION CHAIRMAN: E. W. CUMMINS

Fundamental Characteristics of Pulverized Coal Fly Ashes—L. J. Minnick, presented by the author.

Studies of Limestone Aggregates by Fluid-Flow Methods—W. L. Dolch, presented by the author.

Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction—L. Pepper and Bryant Mather, presented by Mr. Mather.

Control Testing for Separation of Lightweight Materials from Aggregate—E. C. Higginson and G. B. Wallace, presented by Mr. Higginson.[3]

(Continued in the Fifth Session)

SYMPOSIUM ON RADIOISOTOPES IN METALS ANALYSIS AND TESTING[4]

MONDAY, JUNE 22, 2:00 P.M. AND 8:00 P.M.

Sponsored by Committee E-3 on Chemical Analysis of Metals

SESSION CHAIRMAN: W. W. MEINKE

This symposium was organized to afford an opportunity to discuss different applications of radioisotopes to the metals industry. Radioisotopes do not hold the answer to all analysis problems! They too have their limitations. But there are a number of areas where they furnish unexcelled versatility and adaptability. It was the purpose of this symposium to provide a forum for the presentation and discussion of information in these areas.

General Session

Introduction—H. F. Beeghly.

Nucleonics in Analysis—W. W. Meinke.

Instrumentation and Techniques for Analysis with Radioisotopes—A. H. Emmons.

Metals Analysis by Radioactivation—G. H. Morrison.

Refereeing by Isotope Dilution Methods—C. Rosenblum.

Applications in Analysis

Neutron Activation Analysis of Traces of Molybdenum in Tungsten—J. F. Cosgrove.

Application of the (n, alpha) Reaction—A. H. Bushey.

Experiences with Activation in the Analysis of Aluminum—J. E. Lewis.

Training Industrial Personnel in Radioisotope Utilization—J. P. Danforth.

General Panel Discussion

Panel consisted of all speakers, with W. W. Meinke as moderator.

---

[3] Published in the ASTM BULLETIN, No. 243, Jan., 1960, p. 60.

[4] Issued as separate publication *ASTM STP No. 261.*

FOURTH SESSION—COMMITTEE REPORT SESSION

MONDAY, JUNE 22, 4:30 P.M.

SESSION CHAIRMAN: G. H. HARNDEN

Committee B-1 on Wires for Electrical Conductors:

Report presented in the absence of the chairman by C. E. Topping, vice-chairman, and the following actions taken:

Accepted as Tentative:

Test for Electrical Conductivity by Use of Eddy Currents (B 342 – 59 T)
The editorial changes which were made in the title of the test as preprinted are reflected in the above wording.
Specs. for Aluminum-Coated (Aluminized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 341 – 59 T)

Accepted as Tentative, Revisions in:

Spec. for Concentric-Lay-Stranded Aluminum Conductors, Steel Reinforced (ACSR) (B 232 – 58 T), with the following additional change:
Section 1(a).—Change by the addition in line 3 of the words "or aluminum-coated" after "zinc-coated."
Spec. for Soft or Annealed Coated Copper Conductors for Use in Hookup Wire for Electronic Equipment (B 286 – 57 T)

Adopted as Standard, Revisions in:

Spec. for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 56)
Spec. for Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 – 58)
Test for Resistivity of Electrical Conductor Materials (B 193 – 58)
Spec. for Concentric-Lay-Stranded Aluminum Conductors, Hard-Drawn, Three-Quarter Hard-Drawn and Half-Hard Drawn (B 231 – 58)
Spec. for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 245 – 58)
Spec. for Zinc-Coated (Galvanized) Steel Core Wire (With Coatings Heavier Than Standard Weight) for Aluminum Conductors, Steel Reinforced (ACSR) (B 261 – 58)

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented in the absence of the chairman by A. W. Tracy, secretary, and accepted as a report of progress.

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented by C. H. Sample, chairman, and the following action taken:

Adopted as Standard, Revisions in:

Spec. for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 – 58)

Committee B-9 on Metal Powders and Metal Powder Products:

Report presented by J. L. Bonanno, chairman, and the following actions taken:

Accepted as Tentative:

Test for Hardness of Sintered Metal Friction Materials (B 347 – 59 T)
Spec. for Machinable High-Density Tungsten-Nickel-Copper Alloys (B 346 – 59 T)

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices:

Report presented in the absence of the chairman by F. Y. Speight, ASTM staff, and the following actions taken:

Accepted as Tentative:

Specs. for Miniature Electron Tube Leads (F 10 – 59 T)

Adopted as Standard, Revisions in:

Method for Bend Testing of Wire (Wire for Radio Tubes and Incandescent Lamps) (F 113 – 41)
Method of Testing Fine Round and Flat Wire for Electronic Devices (F 219 – 50)

Standards Withdrawn:

Spec. for Round Chromium-Copper Wire for Electronic Devices (F 268 – 56)
Methods of Testing Wire for Supports Used in Electronic Devices and Lamps (F 157 – 50)

FIFTH SESSION—CONCRETE

(*Continued from the Third Session*)

MONDAY, JUNE 22, 8:00 P.M.

SESSION CHAIRMAN: WILLIAM LERCH

Concrete for Shielding Nuclear Radiations—H. S. Davis, presented by the author.

An In-Place Strength Test for Low Density Concrete—I. A. Benjamin and G. D. Ratliff, presented by Mr. Benjamin.[5]

Stresses in Deep Beams Subjected to Central and Third Point Loading—M. E. Raville and F. J. McCormick, presented by Mr. McCormick.

Effect of Length to Diameter Ratio on Compressive Strength—An ASTM Cooperative Investigation—C. E. Kesler, presented by the author.

---

[5] Published in the ASTM BULLETIN, No. 241, Oct., 1959, p. 23.

SIXTH SESSION—SYMPOSIUM ON TIME RATES OF LOADING IN SOIL TESTING[6]

MONDAY, JUNE 22, 8:00 P.M.

SESSION CO-CHAIRMEN: D. M. BURMISTER
L. E. GREGG

Dynamic and Static Resistance of Cohesive Soil—1846 to 1958—W. S. Housel, presented by the author.

The Effect of Rate of Strain on Effective Stresses in Sensitive Clay—C. B. Crawford, presented by the author.

A Method for Adjusting Strain-Rates to Obtain Pore Pressure Measurements in Triaxial Shear Tests—W. Ellis and W. G. Holtz, presented by Mr. Holtz.

Apparatus for Testing Soils Under Repeated Loading—H. B. Seed and J. W. N. Fead, presented by Mr. Raymond Lundgren.

Strain-Rate Behavior of Clay and Organic Solids—D. Burmister, presented by the author.

Automation in Consolidation Testing—R. H. Karol, presented by title.

Time Effects on the Consolidation of Clays—G. A. Leonards and B. K. Ramiah, presented by Mr. Leonards.

The Use of Visco-Elastic Stress-Strain Laws in Soil Testing—R. L. Schiffman, presented by the author.

Discussion.

---

[6] Issued as separate publication *ASTM STP No. 254.*

SEVENTH SESSION—FATIGUE

MONDAY, JUNE 22, 8:00 P.M.

SESSION CO-CHAIRMEN: R. E. PETERSON
L. J. GROVER

Tension, Compression, and Fatigue Properties of Several Steels for Aircraft Bearing Applications—G. Sachs, R. Sell, and W. F. Brown, Jr., presented by Mr. W. W. Gerberich.

The Effect of Temper-Embrittlement on the Fatigue Properties and Damage Due to Fatigue Overstressing of a 3140 Steel—E. B. Mikus and C. A. Siebert, presented by Mr. Mikus.

The Effect of Stress Cycling on the Static Mechanical Properties of SAE 4340 Steel—J. Marin, P. Borachia, and U. Rimrott, presented by Mr. Rimrott.

Some Observations on the Plastic Work Required to Fracture Stainless Steel Under Cyclic Loading—D. E. Martin and Jule Brinn, presented by Mr. Martin.

(*Continued in the Eighth Session*)

EIGHTH SESSION—FATIGUE

(Continued from the Seventh Session)

Tuesday, June 23, 9:30 a.m.

Session Chairman: F. M. Howell

Techniques and Equipment for Fatigue Testing at Very High Frequencies—E. A. Neppiras, presented by Mr. A. Q. Mowbray.

A Method for Determining the Fatigue Limit of Metals by Means of Stepwise Load Increase Test—N. Enomoto, presented by Mr. H. T. Corten.

The Effect of Internal Heating on the Fatigue Life of Titanium—J. P. Romualdi and E. D'Appolonia, presented by Mr. Romualdi.

Anisotropy of Crack Initiation and Propagation in Cold Rolled Aluminum Sheet—H. A. Lipsitt, F. W. Forbes, and R. B. Baird, presented by Mr. Lipsitt.

Tensile and Fatigue Properties of Laminate Sheet Structures—R. B. Baird, F. W. Forbes, and H. A. Lipsitt, presented by Mr. Baird.

NINTH SESSION—SOILS[7]

Tuesday, June 23, 9:30 a.m.

Session Chairman: W. H. Goetz

Laboratory and Field Tests of Cement-Treated Granular Base-Course Materials—M. S. Abrams, presented by the author.

Powder *Versus* Slurry Application of Lime for Soil Stabilization—D. T. Davidson, G. Noguera, and J. B. Sheeler, presented by Mr. Sheeler.

Improved Determination of Preconsolidation Pressure of a Sensitive Clay—J. J. Hamilton and C. B. Crawford, presented by Mr. Crawford.

Technical Session on Electron Metallography[8]

Tuesday, June 23, 9:00 a.m.

Sponsored by Subcommittee XI on Electron Metallography of Metals of Committee E-4 on Metallography

Session Chairman: Anna M. Turkalo

Effect of Heat Treatment on a Cast Nickel Alloy—C. G. Bieber and R. F. Decker.

Morphology of Phases in High-Temperature Alloys as Revealed by the Electron Microscope—J. R. Mihalisin.

Simplified Electron Metallography of Steels—G. E. Pellissier.

Techniques for Studying the Structure and Growth of Tin Oxide by Electron Microscopy—P. S. Trozzo.

Electron Metallography of Neutron-Irradiated Steels—R. F. McCartney.

Electron Microscopy of Tin Plate Alloy Layer—P. A. Stoll.

Electron Microscope Observations of Dislocations in Thin Metal Foils—R. M. Fisher and A. Szirmae.

Subcommittee XI Progress Report on Electron Microstructure of Precipitation-Hardenable Austenitic and Nickel-Base Alloys—R. M. Slepian.

Instrumental Modifications for Routine Electron Diffractometry—G. R. Grieger.

Structure Analysis with Routine Electron Diffraction—G. R. Grieger.

Composition of γ'^2 Phase in High Temperature Alloys from Electron Microprobe Measurements—L. S. Birks and R. E. Seebold.

A Simple Application of the Carbon Replica Technique for Electron Metallography—L. Pellier.

---

[7] These papers are included in *ASTM STP No. 254.*

[8] Issued as a separate publication *ASTM STP No. 262.*

TENTH SESSION—LUNCHEON, PRESIDENT'S ADDRESS, INTRODUCTION OF NEW OFFICERS, RECOGNITION OF 40-YEAR AND 50-YEAR MEMBERS, HONORARY MEMBERSHIPS, REPORT OF BOARD OF DIRECTORS, AWARDS

TUESDAY, JUNE 23, 12:00 NOON

SESSION CHAIRMAN: NATIONAL DIRECTOR CHARLES R. STOCK

About 375 were present at this annual luncheon session, including a number of ladies and visitors.

Awards of Merit:

The Chairman of the 1959 Award of Merit Committee, Past-President H. L. Maxwell, read brief citations and presented the following to President K. B. Woods, who conferred on them, on behalf of the Board of Directors, the Certificate of Award of Merit:

Victor E. Grotlisch
George O. Hiers
Elmer G. Kimmich
Bryant Mather
Mary R. Norton
*Lewis S. Reid
Claude K. Rice
Henry J. Schweim
Benoit J. Sirois
Charles K. Strobel
Herbert W. Stuart
Beaumont Thomas
George L. Werley

---

* Presented in absentia. Deceased July 15, 1959.

Election of Officers:

Results of the letter ballot on election of new officers were announced by W. E. Chalfant on behalf of the Teller's Committee consisting of W. F. Bartoe, Chairman, and Mr. Chalfant. The results were as follows:

For President, to serve for one year:
  F. L. LaQue, 2332 votes.
For Vice-President, to serve for two years:
  M. N. Clair, 2318 votes.
For Directors, to serve for three years:
  A. B. Cornthwaite, 2229 votes.
  C. L. Kent, 2216 votes.
  H. C. Miller, 2210 votes.
  C. F. Nixon, 2210 votes.
  H. D. Wilde, 2192 votes.
  I. V. Williams, 2191 votes.
For Directors, to serve for two years:
  R. D. Thompson, 2276 votes.

The newly-elected members of the Board of Directors who were present were introduced, as were President-elect LaQue and Vice-President-elect Clair, and A. Allan Bates, whose term as Vice-President continues another year.

Passing of the Gavel:

The gavel of authority was presented to Vice-President LaQue by President Woods although the actual shift in authority does not take place until the close of the Annual Meeting. In turn, Mr. LaQue presented to Mr. Woods a certificate, pin, and gavel for his year of service to the Society as its President.

Recognition of Retiring Past-President and Directors:

President Woods presented framed certificates to those retiring from the Board of Directors. Mention was made that this meeting brings to a close for C. H. Fellows nine years on the Board of Directors, as Director, Vice-President, President, and Past-President. Recognition was also given to H. C. Cross, G. H. Harnden, R. R. Litehiser, and C. R. Stock as retiring Directors.

Award of Honorary Memberships:

The Executive Secretary read citations for the following two members who had been elected by the Board of Directors to Honorary Membership in the Society:

Douglas E. Parsons
Jerome Strauss

The President then presented to them certificates of Honorary Membership.

Presidential Address:

The annual President's Address was presented by K. B. Woods, on the subject "The Impact of Science on ASTM."[9]

Report of the Board of Directors:

R. J. Painter, Executive Secretary, presented a brief résumé of the Report of the Board of Directors.

Recognition of 40-Year and 50-Year Members:

Chairman Stock called upon President Woods to present certificates to Messrs. Everett W. Boughton and Joseph Brobston, the two 50-year personal members. Chairman Stock read the names of the five companies who had been continuously affiliated with the Society for 50 years and then read the names of the 44 members, both individual and company, who had been continuously affiliated with the Society for 40 years, and certificates to that effect were presented.

---

[9] See p. 56.

ELEVENTH SESSION—STEEL

TUESDAY, JUNE 23, 2:30 P.M.

SESSION CHAIRMAN: PAST-PRESIDENT N. L. MOCHEL

AISI Type 304L Stainless Steel with Improved Strength—R. R. Brady, presented by the author.

Effect of Cold Rolling and Stress Relief on the Sharp Edge Notch and Smooth Tensile Characteristics of AISI 301 and AISI 304L Sheet—G. B. Espey, A. J. Repko, and W. F. Brown, Jr., presented by Mr. Brown.

Relationships Between Composition and Properties of Austenitic Cr-Mn-C-N Stainless Steels—A. Kasak, C. M. Hsiao, and E. J. Dulis, presented by Mr. Kasak.

Property Relationships of Some Cast and Forged Cr-Mn-Ni-N Steels Containing 18 per cent Chromium—K. Haefner, A. F. Lahr, W. L. Meinhart, and J. J. Kanter, presented by Mr. Kanter.

Effect of Composition and Processing Variables on Some Mechanical Properties of Large Ni-Mo-V Rotor Forgings—K. H. Kramer and J. E. Steiner, presented by Mr. Kramer.

TWELFTH SESSION—SYMPOSIUM ON METHODS OF TEST FOR DESIGN OF BITUMINOUS PAVING MIXTURES[10]

TUESDAY, JUNE 23, 2:30 P.M.

SESSION CHAIRMAN: A. B. CORNTHWAITE

General Factors in the Design of Bituminous Paving Mixtures (Introductory Paper)—L. F. Rader, presented by the author.

Theory and Application of a Gyratory Testing Machine for Hot-Mix Bituminous Pavement—J. L. McRae and C. R. Foster, presented by Mr. McRae.

Application of Marshall Method in Hot Mix Design—H. L. Lehmann and V. Adam, presented by Mr. J. M. Rice.

Control of Bituminous Shoulder Construction for the Northern Illinois Toll Highway—J. J. Waddell, presented by the author.

(*Continued in the Seventeenth Session*)

---

[10] Issued as separate publication *ASTM STP No. 252.*

THIRTEENTH SESSION—COMMITTEE REPORT SESSION

TUESDAY, JUNE 23, 4:30 P.M.

SESSION CHAIRMAN: VICE-PRESIDENT F. L. LAQUE

Committee A-6 on Magnetic Properties:

Report presented by A. C. Beiler, chairman, and accepted as a report of progress.

Committee A-7 on Malleable-Iron Castings:

Report presented by W. M. Albrecht, chairman, and accepted as a report of progress.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

Report presented by L. L. Wyman, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Corrosion-Resisting Chromium Steel Clad Plate, Sheet, and Strip (A 263 – 44 T)
Spec. for Corrosion-Resisting Chromium-Nickel Steel Clad Plate, Sheet, and Strip (A 264 – 44 T)
Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 58 T) (Joint with Committee A-1)

Adopted as Standard:

Spec. for Hot-Rolled and Cold-Finished Corrosion-Resisting Steel Bars (A 276 – 57 T)

Adopted as Standard, Revisions in:

Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 58) (Joint with Committee A-1)
Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 58) (Joint with Committee A-1)
Spec. for Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 – 55), with a change in phosphorus and sulfur content of grade CA-40 from "0.04" to "0.05" per cent max.
Spec. for Heat-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 297 – 55)

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by W. H. Jennings, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 – 59)
Spec. for Phosphor Bronze Plate, Sheet, Strip, and Rolled Bar (B 103 – 55)

The revision of Specifications B 103 was erroneously indicated in the preprinted report for immediate adoption.

Adopted as Standard:

Spec. for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip, and Rolled Bar (B 248 – 58 T)
Spec. for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (B 249 – 58 T)
Spec. for Seamless Copper Tube for Refrigeration Field Service (B 280 – 58 T)
Spec. for Tellurium Copper Rod (B 301 – 58 T)
Spec. for Copper-Silicon Alloy Seamless Pipe and Tube (B 315 – 58 T)
Spec. for Copper-Nickel-Zinc Alloy (Nickel Silver) and Copper-Nickel Alloy Plate, Sheet, Strip, and Rolled Bar (B 122 – 55 T), with the following additional changes:

Section 1.—Change the zinc content for alloy No. 8 from "31.5" to "29" per cent.

Tables I and II.—Change alloy No. 8 to read "(59.0 Cu, 12.0 Ni, 29.0 Zn)."

Spec. for General Requirements for Copper and Copper-Alloy Pipe and Tube (B 251 – 58 T)

Adopted as Standard, Revisions in:

Spec. for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 54)
Spec. for Seamless Copper Tube (B 75 – 58)
Spec. for Threadless Copper Pipe (B 302 – 58)
Spec. for Copper Drainage Tube (DWV) (B 306 – 58)
Spec. for Seamless Copper Bus Pipe and Tube (B 188 – 58)

The recommendation for the revision of Specification B 188 was inadvertently omitted from the preprinted report. The revision relates to Table IV and consists of adding for Type M additional requirements as indicated in the report for Table II of Specification B 251.

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by W. Babington, chairman, and accepted as a report of progress.

Committee C-3 on Chemical-Resistant Mortars:

Report presented in the absence of the chairman by W. C. Hansen, and the following actions taken:

Accepted as Tentative:

Test for Chemical Resistance of Mortars (C 267 - 59 T)

Accepted as Tentative, Revisions in:

Rec. Practice for Installing Sulfur Mortar Joints (C 386 - 57 T)
Rec. Practice for Use of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 397 - 57 T)
Rec. Practice for Use of Resin-Type Chemical-Resistant Mortars (C 399 - 57 T)

The committee withdrew from the report as preprinted the recommendation for the revision of Tentative Recommended Practice for Use of Hydraulic Cement Mortars in Chemical-Resistant Masonry (C 398 - 57 T)

Committee C-4 on Clay Pipe:

Report presented in the absence of the chairman by C. R. Velzy, vice-chairman, and accepted as a report of progress.

Committee C-8 on Refractories:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Accepted as Tentative:

Tests for Size and Bulk Density of Insulating Fire Brick (C 437 - 59 T)
Test for Reheat Change of Carbon Brick and Shapes (C 436 - 59 T)
Test for Thermal Conductivity of Plastic Refractories (C 438 - 59 T)
Test for Resistance to Thermal Spalling of Silica Brick (C 439 - 59 T)
Specs. for Steel Pouring Pit Refractories (C 435 - 59 T)
Specs. for Insulating Fire Brick for Linings of Industrial Furnaces Operated with a Neutral or Oxidizing Atmosphere (C 434 - 59 T)

Accepted as Tentative, Revisions in:

Method of Testing Refractory Brick Under Load at High Temperatures (C 16 - 49)
Test for Reheat Change of Refractory Brick (C 113 - 46)
Def. of Terms Relating to Refractories (C 71 - 55)

Tentative Revision Withdrawn:

Method for Basic Procedure in Panel Spalling Test for Refractory Brick (C 38 - 58)

FOURTEENTH SESSION—MARBURG LECTURE,[11] MEDAL AWARDS

TUESDAY, JUNE 23, 5 P.M.

SESSION CHAIRMAN: PRESIDENT K. B. WOODS

Charles B. Dudley Medal Award:

The Twenty-ninth Charles B. Dudley Medal was made to R. J. MacDonald, Senior Metallurgist, Clevite Research Center, Cleveland, Ohio; R. L. Carlson, Assistant Division Chief, Battelle Memorial Institute, Columbus, Ohio; and W. T. Lankford, Chief Research Engineer, Specialty Products, United States Steel Corp., Monroeville, Pa., for their paper on "The Effect of Strain Rate and Temperature on the Stress-Strain Relations of Deep-Drawing Steel," published in the 1956 *Proceedings*.

Richard L. Templin Award:

The Thirteenth Richard L. Templin Award was made to J. H. Westbrook, General Electric Research Laboratory, Schenectady, N. Y., for his paper on

---

[11] Issued as a separate publication; also ASTM BULLETIN, No. 245, April, 1960.

"Microhardness Testing at High Temperatures," published in the 1957 *Proceedings*.

Sam Tour Award:

The Eighth Sam Tour Award was made to John B. Rittenhouse, Research Group Supervisor, Chemistry Section, Jet Propulsion Laboratory, California Institute of Technology, Pasadena, Calif., for his paper on "The Corrosion and Ignition of Titanium in Fuming Nitric Acid," published in the Symposium on Titanium, *ASTM STP 204*.

Edgar Marburg Lecture:

A brief statement of the purpose of the lecture was given by President Woods. Herman F. Mark, Director, Polymer Research Institute, Polytechnic Institute of Brooklyn, was then introduced by President Woods as the thirty-third Edgar Marburg Lecturer. Speaking on the subject "New Polymers—New Problems," Mr. Mark discussed the fact that during the past few years new catalytic systems have been discovered and new methods of polymerization have been developed which permit the preparation of many organic polymers with a much higher degree of molecular regularity than was possible before. Some of these new materials have already found interesting and meaningful applications. Others are just now being investigated for profitable and promising uses. In addition to their practical applicability, these highly ordered macromolecules have also posed new fundamental questions of nomenclature, classification, and characterization. They force us to widen our scope and to expand the basic framework of polymer science so that we can adequately describe and interpret all the data assembled within the past few years.

President Woods expressed appreciation to Mr. Mark for his excellent lecture and presented to him on behalf of the Society the Edgar Marburg Lecture Certificate.

FIFTEENTH SESSION—EFFECT OF TEMPERATURE

TUESDAY, JUNE 23, 8:00 P.M.

SESSION CHAIRMAN: H. C. CROSS

Joint Committee on Effect of Temperature on the Properties of Metal:

Report presented by J. J. Kanter, chairman, and accepted as a report of progress.

Papers:

Creep-Rupture Behavior of Notched and Unnotched Specimens of Types 304, 316 and 321 Austenitic Stainless Steels—F. Garofalo, presented by the author.

Effect of Environment on Creep and Creep-Rupture Behavior of Several Steels—F. Garofalo, presented by the author.

Effect of Notch Geometry on the Tensile Properties of Annealed Titanium at 100, 25, −78, and −196 C—G. W. Geil and N. L. Carwile, presented by Mr. Geil.

Stress-Stain Properties of Selected Titanium—E. P. Klier and C. Gazzara, presented by Mr. Klier.

Properties of 70–30 Copper-Nickel Alloy at Temperatures Ranging up to 1050 F—W. F. Simmons, R. I. Jaffe, D. N. Williams and B. J. Sirois, presented by Mr. Simmons.

Mechanical Properties of a High Purity Lead and a 0.058 per cent Copper-Lead Alloy at Elevated Temperatures—T. E. Tietz, presented by the author.

SIXTEENTH SESSION—SYMPOSIUM ON ATTERBERG LIMITS[6]

TUESDAY, JUNE 23, 8:00 P.M.

SESSION CHAIRMAN: G. F. SOWERS

History and Development of the Atterberg Limits Tests—E. E. Bauer, presented by the author.

The Use of a One-Point Liquid Limit Procedure—W. J. Eden, presented by Mr. Legget.

Ohio Adopts the One-Point Mechanical Method for Determining the Liquid Limit of Soils—J. G. Joslin and H. D. Davis, presented by Mr. Joslin.

Correlation of Atterberg Limits with Geology for Deep Cores from Subsidence Areas in California—A. I. Johnson and D. A. Morris, presented by Mr. W. G. Holtz.

Investigations of the Liquid Limit Test on Soils—R. F. Dawson, presented by the author.

Liquid Limit Results from Various Types of Grooving Tools—J. E. Mitchell, presented by the author.

Proposed Universal Standards for the Liquid Limit Tests—M. D. Morris, R. B. Ulp, and R. A. Spinna, presented by Messrs. Morris and Ulp.

Plastic-Limit—Comparison of Cube and Standard Test Methods—E. A. Abdun-Nur, presented by the author.

Penetration Tests for Liquid Limit—G. F. Sowers, A. Vesic, and M. Grandolfi, presented by Mr. Sowers.

SEVENTEENTH SESSION—SYMPOSIUM ON METHODS OF TEST FOR DESIGN OF BITUMINOUS PAVING MIXTURES[10]

(Continued from the Twelfth Session)

TUESDAY, JUNE 23, 8:00 P.M.

SESSION CHAIRMAN: L. F. RADER

Triaxial Testing of Bituminous Mixtures—W. H. Goetz and J. H. Schaub, presented by Mr. Goetz.

Correlation of Hveem Stabilometer and Cohesiometer Test Results and Kneading Compactor Densities with Service Records of Bituminous Pavements—C. E. Minor, presented by Mr. L. F. Rader.

Void Requirements for Dense Graded Bituminous Paving Mixtures—N. W. McLeod, presented by the author.

The Use of the Immersion-Compression Test in Evaluating and Designing Bituminous Paving Mixtures—J. F. Goode, presented by the author.

EIGHTEENTH SESSION—GENERAL TESTING

WEDNESDAY, JUNE 24, 9:30 A.M.

SESSION CHAIRMAN: A. H. KIDDER

Development of ASTM Standard Reference Blocks for Ultrasonic Inspection—F. C. Panian and H. E. Van Valkenburg, presented by Mr. Panian.

A Cooperative Study of the Hardness Testing of Cemented Carbides—B. M. Caugherty, H. T. Oatman, and O. W. Reen, presented by Mr. Reen.

New Joint Sealer Testing Machine Developed—R. J. Schutz and A. Van Hauter, presented by Mr. Schutz.[12]

Experimental Observations on the Relation Between Polarization Resistance and Corrosion Rate—M. Stern and E. D. Weisert, presented by Mr. W. Weisenberg.

(Continued in the Twenty-First Session)

---

[12] Published in the ASTM BULLETIN, No. 241, Oct., 1959, p, 42.

NINETEENTH SESSION—SYMPOSIUM ON SPECTROSCOPIC EXCITATION[13]

WEDNESDAY, JUNE 24, 9:30 A.M.

SESSION CHAIRMAN: B. F. SCRIBNER

Introductory Remarks—B. F. Scribner, presented by the author.

The Matrix Effect in Excitation—J. K. Hurwitz, presented by the author.

Prepared Discussions, F. R. Potter and L. O. Eikren.

Effects of Gaseous Atmospheres on Excitation—G. Andermann and J. W. Kemp, presented by Mr. Kemp.

Prepared Discussions, V. A. Fassel and T. P. Schreiber.

Some Properties of New or Modified Excitation Sources—M. Margoshes, presented by the author.

Prepared Discussions, F. Brech and J. T. Rozsa.

---

[13] Issued as separate publication *ASTM STP No. 259.*

THE PLACE OF RESEARCH IN ASTM

(An Open Forum for Technical Committee Officers)

WEDNESDAY, JUNE 24, 9:00 A.M.

Sponsored by Administrative Committee on Research

Promotion of knowledge of materials through research and by other means has traditionally held equal position in the Society with development of standard test methods and specifications. This aspect of the Society's work is correlated by the Administrative Committee on Research. The committee sponsored this Forum to provide an opportunity to discuss the various aspects of the subject and to consider how the Technical Committees and the Administrative Committee can best cooperate in the promotion and development of research.

TWENTIETH SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 24, 11:15 A.M.

SESSION CHAIRMAN: E. C. SHUMAN

Committee C-13 on Concrete Pipe:

Report presented by R. R. Litehiser, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Joints for Circular Concrete Sewer and Culvert Pipe Using Flexible, Watertight, Rubber-Type Gaskets (C 443 - 59 T)

Specs. for Perforated Concrete Pipe (C 444 - 59 T)

Accepted as Tentative, Revisions in:

Spec. for Reinforced Concrete Culvert, Storm Drain, and Sewer Pipe (C 76 - 57 T)

Spec. for Reinforced Concrete Low-Head Pressure Pipe (C 361 - 57 T)

Spec. for Concrete Pipe for Irrigation or Drainage (C 118 - 56)

Adopted as Standard, Revisions in:

Spec. for Concrete Sewer Pipe (C 14 - 58)

Spec. for Concrete Pipe for Irrigation or Drainage (C 118 - 56)

Tentative Withdrawn:

Spec. for Reinforced Concrete Low Head Internal Pressure Sewer Pipe (C 362 - 55 T)

Committee C-16 on Thermal Insulating Materials:

Report presented in the absence of the chairman by E. C. Shuman, and the following actions taken:

Accepted as Tentative:

Test for Normal Total Emittance of Surfaces of Materials 0.01 in. or Less in Thickness at

Approximately Room Temperature (C 445 – 59 T)
Method for Determining the Maximum Use Temperature of Preformed High-Temperature Insulation (C 447 – 59 T)
Test for Breaking Strength and Calculated Modulus of Rupture of Preformed Insulation for Pipes (C 446 – 59 T)

Accepted as Tentative, Revisions in:

Spec. for Diatomaceous Earth Block-Type Thermal Insulation (C 333 – 54 T)
Spec. for Diatomaceous Earth Thermal Insulation for Pipes (C 334 – 54 T)
Tests for Water Vapor Transmission of Materials Used in Building Construction (C 355 – 54 T)
Spec. for 85 per cent Magnesia Thermal Insulating Cement (C 193 – 48) (Standard Reverted to Tentative)
Spec. for Expanded or Exfoliated Vermiculite Thermal Insulating Cement (C 196 – 48) (Standard Reverted to Tentative)
Spec. for Diatomaceous Silica Thermal Insulating Cement (C 197 – 48) (Standard Reverted to Tentative)

Committee C-19 on Structural Sandwich Constructions:

Report presented in the absence of the chairman by E. C. Shuman, and accepted as a report of progress.

Committee C-20 on Acoustical Materials:

Report presented in the absence of the chairman by E. C. Shuman, and accepted as a report of progress.

Committee C-21 on Ceramic Whitewares and Related Products:

Report presented in the absence of the chairman by E. C. Shuman, and accepted as a report of progress.

Committee C-22 on Porcelain Enamel:

Report presented in the absence of the chairman by E. C. Shuman, and the following actions taken:

Accepted as Tentative:

Tests for Abrasion Resistance of Porcelain Enamels (C 448 – 59 T)

Adopted as Standard:

Test for 45-deg Specular Gloss of Ceramic Materials (C 346 – 55 T)

Adopted as Standard, Revisions in:

Test for Adherence of Porcelain Enamel and Ceramic Coatings to Sheet Metal (C 313 – 55)

Committee E-5 on Fire Tests of Materials and Construction:

Report presented by W. J. Krefeld, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Defining Noncombustibility of Building Materials (E 136 – 58 T)
Fire Hazard Classification of Building Materials (E 84 – 50 T)
Fire Tests of Building Construction and Materials (E 119 – 58)

Committee E-6 on Methods of Testing Building Constructions:

Report presented by R. F. Legget, chairman, and accepted as a report of progress.

HIGHWAY MATERIALS LUNCHEON AND AWARDS

WEDNESDAY, JUNE 24, 12:00 NOON

TOASTMASTER: HARMER E. DAVIS

The luncheon was sponsored by Committees A-1 on Steel, C-1 on Cement, C-9 on Concrete and Concrete Aggregates, D-4 on Road and Paving Materials, and D-18 on Soils for Engineering Purposes.

The guest speaker was Ellis L. Armstrong, U.S. Commissioner of Public Roads, whose subject was "Research—A Forward Look."[14]

---

[14] Published in the ASTM BULLETIN, No. 241, Oct., 1959, p. 14.

C. A. Hogentogler Award:

The Fourth C. A. Hogentogler Award was made to Werner E. Schmid, Assistant Professor, Department of Civil Engineering, Princeton University, Princeton, N. J., for his paper "The Permeability of Soils and the Concept of a Stationary Boundary-Layer," published in the 1957 *Proceedings*.

Sanford E. Thompson Award:

The Fifteenth Sanford E. Thompson Award was made to George Verbeck, Manager, Applied Research Section, Portland Cement Assn., Chicago, Ill., for his paper on "Carbonation of Hydrated Portland Cement," published in *ASTM STP 205*.

TWENTY-FIRST SESSION—GENERAL TESTING

(Continued from the Eighteenth Session)

WEDNESDAY, JUNE 24, 2:30 P.M.

SESSION CHAIRMAN: J. P. ROMUALDI

A Preliminary Report on Sharp Edge Notch and Smooth Tensile Characteristics of Ultra High Strength Steel Sheet Alloys—G. B. Espey, M. H. Jones, and W. F. Brown, Jr., presented by Mr. Brown.

Experimental Determination of Energy Release Rate for Notch Bending and Notch Tension, and Applications to Fracturing—J. D. Lubahn, presented by the author.

The Tensile Properties of Some Engineering Materials at High Rates of Strain—A. L. Austin and R. F. Steidel, Jr., presented by Mr. Austin.

Compression Testing at Constant True Strain Rates—J. E. Hockett, presented by the author.

Effect of Specimen Taper on the Determination of Elongation in the Tensile Test—E. B. Kula and F. R. Larson, presented by Mr. Kula.[15]

---

[15] Published in ASTM BULLETIN, May, 1959, pp. 58–61.

TWENTY-SECOND SESSION—SYMPOSIUM ON PRACTICAL AND STATISTICAL SIGNIFICANCE OF TESTS AND PROPERTIES OF BITUMINOUS BINDERS[10]

WEDNESDAY, JUNE 24, 2:30 P.M.

SESSION CHAIRMAN: D. F. FINK

Introduction—D. F. Fink, presented by the author.

The Evolution of ASTM Tests and Specifications for Asphaltic Materials—G. Abson, presented by the author.

Practical Significance of Tests on Asphalt Cements—N. W. McLeod, presented by the author.

Precision of Present ASTM Tests on Bituminous Paving Binders—A. B. Brown, presented by the author.

Fundamental Statistical Concepts in Testing—P. E. Irick, presented by the author.

Summary—Possibilities for Extension and Improvement of ASTM Tests on Bituminous Paving Binders—D. F. Fink, presented by the author.

TWENTY-THIRD SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 24, 4:00 P.M.

SESSION CHAIRMAN: I. V. WILLIAMS

Committee A-1 on Steel:

Report presented by J. J. Kanter, chairman, and the following actions taken:

Accepted as Tentative:

Method and Specification for Ultrasonic Testing and Inspection of Steel Plates of Firebox and Higher Quality (A 435 – 59 T), with the following additional change:

Section 2.—Add a new paragraph (*a*) to read as follows, relettering the present paragraph (*a*) and subsequent paragraphs accordingly:

(*a*) The inspection shall be carried out in an area reasonably free from hazards and away from operations that interfere with proper functioning of the equipment.

Spec. for Deformed Billet Steel Bars for Concrete Reinforcement with 60,000 psi Minimum Yield Point (A 432 – 59 T)

Spec. for Leaded Carbon Steel Plates of Flange and Firebox Qualities for Fusion Welded Boilers and Other Pressure Vessels (A 433 – 59 T)

Spec. for Quenched and Tempered Alloy Steel Bars, Hot-Rolled or Cold-Finished (A 434 – 59 T), with the following additional changes:

Table I.—For Classes BC and BD, the size breakdown should be as follows:

1½ and under
Over 1½ to 2½
Over 2½ to 4
Over 4 to 7
Over 7 to 9½

Accepted as Tentative, Revisions in:

Spec. for Rail-Steel Bars for Concrete Reinforcement (A 16 – 57 T), with the following change: The revision for Table I should be indicated as a revision for Table II; in the revision of Section 3(*d*), "7" should be changed to "5" per cent.

Spec. for General Requirements for Hot-Rolled and Cold-Finished Carbon and Alloy-Steel Bars (A 29 – 58 T)

Spec. for Welded and Seamless Steel Pipe (A 53 – 58a T)

Spec. for Seamless Steel Boiler Tubes (A 83 – 58 T)

Spec. for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 – 58 T)

Spec. for Hot-Rolled Carbon Steel Bars (A 107 – 58 T)

Spec. for Cold-Rolled Carbon Steel Strip (A 109 – 58 T)

Spec. for Electric-Resistance-Welded Steel Pipe (A 135 – 58 T)

Spec. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 58 T)

Spec. for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for General Service (A 181 – 58 T)

Spec. for Forged or Rolled Alloy Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 58 T)

Spec. for Seamless Alloy Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 – 58 T)

Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 58 T)

Spec. for Factory-Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings (A 234 – 58a T)

Spec. for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 – 58 T)

Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 58 T) (Joint with Committee A-10)

Spec. for Alloy Steel Bolting Materials for Low-Temperature Service (A 320 – 58 T)

Spec. for Quenched and Tempered Carbon Steel Bars (A 321 – 57 T)

Spec. for Seamless and Welded Steel Tubes for Low-Temperature Service (A 334 – 58 T)

Spec. for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 – 55 T)

Spec. for Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 – 58 T)

Spec. for Forged or Rolled Alloy Steel Pipe Flanges, Forged Fittings, and Valves and Parts Specially Heat Treated for High-Temperature Service (A 404 – 57 T)

Spec. for High-Strength Structural Alloy Rivet Steel (A 406 – 57 T)

Spec. for Carbon Steel Chain (A 413 – 57 T)

Spec. for Uncoated Seven-Wire Stress-Relieved Strand for Prestressed Concrete (A 416 – 57 T)

Spec. for Uncoated Stress-Relieved Wire for Prestressed Concrete (A 421 – 58 T)

Spec. for Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service (A 426 – 58 T)

Spec. for High-Strength Billet Steel Bars for Concrete Reinforcement (A 431 – 58 T)
Spec. for Steel Music Spring Wire (A 228 – 51)

Adopted as Standard:

Rec. Practice for Ultrasonic Testing and Inspection of Heavy Steel Forgings (A 388 – 55 T)

Adopted as Standard, Revisions in:

Spec. for General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 – 56)
Spec. for Structural Steel for Ships (A 131 – 58), with the additional change in Table I consisting of changing the maximum permissible carbon content for check analysis from "0.28" to read "0.27" per cent.
Spec. for High-Strength Structural Rivet Steel (A 195 – 57)
Spec. for Welded and Seamless Steel Pipe Piles (A 252 – 55)
Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 58) (Joint with Committee A-10)
Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 58) (Joint with Committee A-10)

Tentatives Withdrawn:

Spec. for Quenched and Tempered Alloy Steel Bars (A 286 – 57 T)
Spec. for Cold-Finished Quenched and Tempered Alloy Steel Bars (A 364 – 57 T)

Committee A-3 on Cast Iron:

Report presented by T. E. Eagan, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Austenitic Gray Iron Castings (A 436 – 59 T)

Accepted as Tentative, Revisions in:

Spec. for Gray Iron Castings for Pressure-Containing Parts for Temperatures up to 650 F (A 278 – 57) (Standard Reverted to Tentative)

Tentative Withdrawn:

Methods of Testing Chilled and White Iron Castings (A 360 – 55 T)

Committee A-5 on Corrosion of Iron and Steel:

Report presented by H. F. Hormann, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for 1.25 oz Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets (A 361 – 58 T)
Spec. for Zinc-Coated Steel Chain-Link Fence Fabric (A 392 – 55 T)

Adopted as Standard:

Rec. Practice for Safeguarding Against Warpage and Distortion During Hot-Dip Galvanizing of Steel Assemblies (A 384 – 55 T)
Spec. for Zinc-Coated Low-Carbon Steel Armor Wire (A 411 – 57 T)
Rec. Practice for Providing High Quality Zinc Coatings (Hot Dip) on Assembled Products (A 385 – 55 T)
Spec. for Zinc Coating (Hot Dip) on Assembled Steel Products (A 386 – 55 T)

Adopted as Standard, Revisions in:

Spec. for Zinc-Coated (Galvanized) "Iron" Telephone and Telegraph Line Wire (A 111 – 52)
Spec. for Zinc-Coated (Galvanized) Steel Tie Wires (A 112 – 58)
Spec. for Zinc (Hot-Galvanized) Coatings on Products Fabricated from Rolled, Pressed, and Forged Steel Shapes, Plates, Bars, and Strip (A 123 – 53)
Rec. Practice for Safeguarding Against Embrittlement of Hot Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A 143 – 46)
Spec. for Zinc Coating (Hot Dip) on Iron and Steel Hardware (A 153 – 53)
Spec. for Zinc-Coated (Galvanized) High Tensile Steel Telephone and Telegraph Line Wire (A 326 – 52)

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented in the absence of the chairman by A. M. Bounds, secretary, and the following action taken:

Adopted as Standard, Revisions in:

Spec. for Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes (B 170 – 47)

Committee E-3 on Chemical Analysis of Metals:

Report presented in the absence of the chairman by H. A. Bright, honorary chairman, and the following actions taken:

Accepted as Tentative:

Methods for Chemical Analysis of Zirconium and Zirconium-Base Alloys (E 146 – 59 T)

Accepted as Tentative, Revisions in:

Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 - 57 T)

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman, and accepted as a report of progress.

TWENTY-FOURTH SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 24, 4:00 P.M.

SESSION CHAIRMAN: J. B. RATHER, JR.

Committee C-7 on Lime:

Report presented in the absence of the chairman by P. J. Smith, ASTM staff, and the following actions taken:

Accepted as Tentative:

Specs. for Quicklime and Hydrated Lime for Hypochlorite Bleach Manufacture (C 433 - 59 T)

Specs. for Pozzolans for Use with Lime (C 432 - 59 T)

Adopted as Standard, Revisions in:

Spec. for Quicklime for Structural Purposes (C 5 - 26)

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by W. H. Price, chairman, and the following actions taken:

Accepted as Tentative:

Test for Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to the Alkali-Aggregate Reaction (C 441 - 59 T)

Specs. for Cotton Mats for Curing Concrete (C 440 - 59 T)

Accepted as Tentative, Revisions in:

Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 56 T)

Specs. for Waterproof Paper for Curing Concrete (C 171 - 58 T)

Methods of Sampling and Testing Fly Ash for Use as an Admixture in Portland Cement Concrete (C 311 - 57 T)

Specs. for Lightweight Aggregates for Structural Concrete (C 330 - 53 T)

Specs. for Lightweight Aggregates for Concrete Masonry Units (C 331 - 53 T)

Reversion to Tentative:

Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified Cube Method) (C 116 - 49)

Adopted as Standard:

Test for Compressive Strength of Molded Concrete Cylinders (C 39 - 56 T)

Test for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading) (C 293 - 57 T)

Adopted as Standard, Revisions in:

Spec. for Concrete Aggregates (C 33 - 57)

Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 - 57)

Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78 - 57)

Test for Specific Gravity and Absorption of Coarse Aggregates (C 127 - 42) (Joint with Committee D-4)

Test for Specific Gravity and Absorption of Fine Aggregate (C 128 - 57) (Joint with Committee D-4)

Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 - 57)

Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 - 57)

Committee C-12 on Mortars for Unit Masonry:

Report presented by R. E. Copeland, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Mortar for Unit Masonry (C 270 - 57 T)

Tentative Withdrawn:

Spec. for Mortar for Reinforced Brick Masonry (C 161 – 44 T)

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Strontium Chromate (D 1649 – 59 T)
Spec. for Basic Lead Silico-Chromate (D 1648 – 59 T)
Test for Phthalic Acid Isomers and Benzoic Acid in Alkyd Resins and Esters (D 1651 – 59 T)
Test for Epoxy Content of Epoxy Resins (D 1652 – 59 T)
Test for Moisture Vapor Permeability of Organic Coating Films (D 1653 – 59 T)
Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments (D 1654 – 59 T)
Method of Sampling and Testing Shellac Varnish (D 1650 – 59 T)
Test for Gas Checking and Draft Test of Varnish Films (D 1643 – 59 T)
Test for Print Resistance of Varnishes (D 1645 – 59 T)

Accepted as Tentative, Revisions in:

Spec. for Heavy Petroleum Spirits (Heavy Mineral Spirits) (D 965 – 48 T)
Test for Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus (D 1310 – 56 T)
Testing Fatty Acids Used in Protective Coatings (D 1467 – 57 T)
Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 58)
Spec. for Raw Tung Oil (D 12 – 55) (Standard Reverted to Tentative)
Methods of Sampling and Analysis of Shellac (D 29 – 57) (Standard Reverted to Tentative)
Methods of Testing Varnishes (D 154 – 58) (Standard Reverted to Tentative)
Methods of Sampling and Testing Aluminum Powder and Paste (D 480 – 51) (Standard Reverted to Tentative)
Spec. for Liquid Paint Driers (D 600 – 43) (Standard Reverted to Tentative)

Adopted as Standard:

Tests for Nonvolatile Content of Varnishes (D 1644 – 59) (New Standard)
Tests for Elasticity or Toughness of Varnishes (D 1642 – 59) (New Standard)
Test for Acid Value of Varnishes (D 1639 – 59) (New Standard)
Test for Reactivity of Paint Liquids (D 479 – 59) (New Standard)
Test for Resistance to Water and Alkali of Dried Films of Varnishes (D 1647 – 59) (New Standard)
Tests for Drying Times of Varnishes (D 1640 – 59) (New Standard)
Test for Exterior Durability of Varnishes (D 1641 – 59) (New Standard)
Test for Flash Point of Drying Oils (D 1393 – 56 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 58)
Spec. for Petroleum Spirits (Mineral Spirits) (D 235 – 39)
Spec. for Isopropyl Alcohol (Isopropanol) (D 770 – 58)
Methods of Chemical Analysis of White Linseed Oil Paints (D 215 – 41)
Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 – 58)

Tentative Withdrawn:

Methods of Testing Varnishes (D 154 – 58 T)

Committee D-3 on Gaseous Fuels:

Report presented in the absence of the chairman by P. J. Smith, ASTM staff, and accepted as a report of progress.

Committee D-8 on Bituminous Materials for Roofing, Waterproofing, and Related Building or Industrial Uses:

Report presented by H. R. Snoke, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Woven Glass Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 1668 – 59 T)
Method for Preparation of Test Panels for Accelerated and Outdoor Weathering of Bituminous Coatings (D 1669 – 59 T)
Test for Failure Endpoint in Accelerated and Outdoor Weathering of Bituminous Materials (D 1670 – 59 T)

Accepted as Tentative, Revisions in:

Rec. Practice for Accelerated Weathering Test of Bituminous Materials (D 529 – 39 T)
Spec. for Asphalt Shingles Surfaced with Mineral Granules (D 225 – 58)

Spec. for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (D 250 – 56)
Spec. for Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs (D 655 – 47)

Adopted as Standard:

Methods of Sampling and Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 – 58 T)
Spec. for Insulating Siding Surfaced with Mineral Granules (D 1226 – 58 T)
Spec. for Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs (D 1227 – 52 T)
Spec. for Woven Burlap Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 1327 – 57 T)

Committee D-18 on Soils for Engineering Purposes:

Report presented in the absence of the chairman by M. D. Catton, and the following actions taken:

Adopted as Standard:

Test for Plastic Limit and Plasticity Index of Soils (D 424 - 54 T)

Standard Withdrawn:

Test for Field Moisture Equivalent of Soils (D 426 - 39)

In the preprinted report there was inadvertently recommended for withdrawal the Test for Centrifuge Moisture Equivalent of Soils (D 425 - 39).

Committee D-25 on Casein and Similar Protein Materials:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following action taken:

Accepted as Tentative:

Method of Sampling Casein and Similar Protein Materials (D 1697 – 59 T)

Committee D-27 on Electrical Insulating Liquids and Gases:

Report presented by F. M. Clark, chairman, and the following actions taken:

Accepted as Tentative:

Test for Sediment and Soluble Sludge in Service-Aged Insulating Oils (D 1698 – 59 T)

Adopted as Standard, Revisions in:

Method for Sampling Electrical Insulating Oils (D 923 – 56)

Withdrawal of Tentative Revision:

Method for Sampling Electrical Insulating Oils (D 923 – 56)

TWENTY-FIFTH SESSION—GILLETT MEMORIAL LECTURE[16]

WEDNESDAY, JUNE 24, 5:00 P.M.

SESSION CHAIRMAN: VICE-PRESIDENT F. L. LAQUE

Eighth Gillett Memorial Lecture:

Vice-President LaQue opened the session by giving a brief statement of the purpose of the lecture. This lecture is sponsored jointly by the ASTM and the Battelle Memorial Institute to perpetuate the memory of Horace W. Gillett, one of America's leading technologists, the first Director of the Institute, and for many years a very active worker in the Society, through the presentation of a lecture on a subject pertaining to the development, testing, evaluation, and application of metals.

Vice-President LaQue called upon B. D. Thomas, President of Battelle Memorial Institute, who introduced John C. Fisher, Metallurgy and Ceramics Research Department, General Electric Co. Mr. Fisher presented the eighth H. W. Gillett Memorial Lecture on the subject "The Role of Dislocations in Plastic Deformation." The lecturer brought out that it is now known that metals and other crystalline materials deform plastically through the motion of dislocations. These imperfections, which are wrinkles in the atomic layers of a crystal, have been observed by a number of experimental techniques, and their motion has been followed during the course of plastic deformation. High strengths can be achieved by eliminating dislocations entirely, or by the more common method of blocking their motion. At present the dislocation concept makes it possible to account, semiquantitatively, for the strength of alloys.

---

[16] Issued as a separate publication.

Vice-President LaQue expressed appreciation to Mr. Fisher for his outstanding lecture and presented to him on behalf of the Society the H. W. Gillett Memorial Lecture Certificate.

ANNUAL DINNER

WEDNESDAY, JUNE 24, 6:30 P.M.

The Philadelphia District, through its Council, acted as the official host at the Cocktail Party and Dinner which was followed by entertainment. Approximately 330 persons attended this function.

TWENTY-SIXTH SESSION—CEMENT

THURSDAY, JUNE 25, 9:30 A.M.

SESSION CHAIRMAN: VICE-PRESIDENT A. A. BATES

An Approach to the Problem of Evaluation of Plasticity and Workability of Pastes and Mortars—A. D. Conrow, presented by the author.

Quantitative Determination of the Four Major Phases in Portland Cements by X-Ray Analysis—S. Brunauer, L. E. Copeland, D. L. Kantro, C. H. Weisé, and E. G. Schulz, presented by Mr. Hubert Woods.

Control of Gypsum in Portland Cement—B. Tremper, presented by Mr. T. B. Kennedy.

Acetate Processed Portland Cement—B. E. Kester, presented by the author.

Cement Reference Laboratory (1929–1959)—J. R. Dise, presented by the author.

TWENTY-SEVENTH SESSION—ROAD AND PAVING MATERIALS[17]

THURSDAY, JUNE 25, 9:30 A.M.

SESSION CHAIRMAN: C. A. CARPENTER

The Use of Capillary Viscometers and Fundamental Viscosity Units as a Substitute for the Saybolt Furol Viscometer for Practical Refinery Control of Cutback Asphalt Production—D. V. Levy, F. E. Fassnacht, R. D. Umbach, G. P. Hibler, and D. C. Gagle, presented by Mr. Levy.

Density Changes in Asphalt Pavement Core Samples—T. C. Hein and R. J. Schmidt, presented by Mr. Hein.

---

[17] These papers are included in *ASTM STP No. 252*.

SESSION ON AGING AND COMPRESSION SET OF POLY(VINYL CHLORIDE)

THURSDAY, JUNE 25, 9:00 A.M.

Sponsored jointly by Committees D-11 on Rubber and Rubber-Like Materials and D-20 on Plastics

Improved Oven Design for Accelerated Aging of Poly(Vinyl Chloride)—Markus Royen.[17a]

The Effect of Die Closure Pressure on Mooney Viscosity Determinations—Norbert Mueller.

[17a] Published in ASTM BULLETIN, No. 243, Jan., 1960, p. 43.

TWENTY-EIGHTH SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 25, 11:15 A.M.

SESSION CHAIRMAN: F. W. REINHART

Committee D-7 on Wood:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Accepted as Tentative:

Methods of Conducting Machining Tests of Wood and Wood-Base Materials (D 1666 - 59 T)

Accepted as Tentative, Revisions in:

Spec. for Wood Paving Blocks for Exposed Platforms, Pavements, Driveways, and Interior Floors Exposed to Wet and Dry Conditions (D 52 - 58 T)

Adopted as Standard, Revisions in:

Test for Coke Residue of Creosote (D 168 - 30)
Test for Distillation of Creosote (D 246 - 49)
Spec. for Creosoted End-Grain Wood Block Flooring for Interior Use (D 1031 - 55)
Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (D 1101 - 58)

Committee D-10 on Shipping Containers:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following action taken:

Accepted as Tentative, Revisions in:

Vibration Test for Shipping Containers (D 999 - 48 T)

Committee D-12 on Soaps and Other Detergents:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Accepted for Publication as Information Only:

Method for Rapid Determination of Solids by Infrared Moisture Balance

Accepted as Tentative:

Test for Synthetic Anionic Active Ingredient in Detergents by Cationic Titration Procedure (D 1681 - 59 T)

Accepted as Tentative, Revisions in:

Methods for Sampling and Analysis of Sodium Triphosphate (D 501 - 58 T)
Methods of Testing Detergent Alkylate (D 1569 - 58 T)

Standards Withdrawn:

Spec. for Palm Oil Solid Soap (D 535 - 52)
Spec. for Palm Oil Chip Soap (D 536 - 52)
Spec. for Olive Oil Solid Soap (D 592 - 42)

Committee D-13 on Textile Materials:

Report presented by B. L. Whittier, chairman, and the following actions taken subject to confirming letter ballot of Committee D-13:

Accepted as Tentative:

Test for Construction Characteristics of Woven Fabrics (D 39 - 59 T), as a tentative revision of Sections 3 and 4 of Standard Methods D 39 - 49.
Methods for Testing Breaking Load and Elongation of Textile Fabrics (D 1682 - 59 T), as a tentative revision of Sections 9 to 14 of Standard Methods D 39 - 49.
Test for Seam Breaking Strength of Woven Textile Fabrics (D 1683 - 59 T)
Rec. Practice for Lighting Cotton Classing Rooms for Color Grading (D 1684 - 59 T)

Accepted as Tentative, Revisions in:

Methods of Identification of Fibers in Textiles (D 276 - 57 T)
Methods of Quantitative Analysis of Textiles (D 629 - 57 T)
Methods of Testing and Tolerances for Tire Cords from Man-Made Fibers (D 885 - 58 T)
Test for Twist in Single Spun Yarns (Untwist-Twist Method) (D 1422 - 58 T)
Test for Twist in Yarns (Direct-Counting Method) (D 1423 - 58 T)
Test for Number of Neps in Cotton Fibers (D 1446 - 53 T)
Test for Dimensional Change of Knit Fabrics (D 1470 - 57 T)
Tests for Pilling Propensity of Textile Fabrics (D 1375 - 55 T)
Test for Unevenness of Textile Strands (D 1425 - 56 T)
Test for Breaking Strength of Yarn by Skein Method (D 1578 - 58 T)
Def. of Terms Relating to Textile Materials (D 123 - 58)

Adopted as Standard:

Test for Man-Made Staple Fibers (D 540 - 57 T)
Test for Relaxation and Felting Shrinkage in Laundering of Stabilized Knit Wool Fabrics (D 1284 - 53 T)
Test for Tear Resistance of Woven Fabrics by Falling-Pendulum (Elmendorf) Apparatus (D 1424 - 56 T)
Spec. and Test for Fineness of Mohair Tops (D 1381 - 57 T)
Test for Non-Lint Content of Cotton (Shirley Analyser Method) (D 1451 - 56 T)

The recommendation for adoption as standard of Tentative Method of Test for Yarn Distortion of Woven Fabrics (D 1336 - 54 T) failed of approval in Committee D-13 and therefore it has been deleted from the report.

Adopted as Standard, Revisions in:

Def. of Terms Relating to Textile Materials (D 123 - 58), further revised by the adoption of tentative definitions for "Ribbon, Slit or Cut," and "Ribbon, Woven."
Spec. and Test for Asbestos Roving for Electrical Purposes (D 375 - 58)
Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics (D 462 - 53)
Methods of Testing Woven Fabrics (D 39 - 49)
Methods of Testing Felt (D 461 - 57)
Methods of Testing Nonwoven Fabrics (D 1117 - 58)
Test for Fineness of Cotton Fibers by Micronaire (D 1448 - 58)

Committee D-14 on Adhesives:

Report presented in the absence of the chairman by J. J. Lamb, secretary, and accepted as a report of progress.

Committee D-15 on Engine Antifreezes:

Report presented by R. E. Vogel, chairman, and the following action taken:

Adopted as Standard, Revisions in:

Test for Water in Concentrated Engine Antifreezes by the Iodine Reagent Method (D 1123 - 54)

Committee D-21 on Wax Polishes and Related Materials:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Adopted as Standard:

Test for Acid Number (Empirical) of Natural Waxes (D 1386 - 55 T)
Test for Saponification Number (Empirical) of Natural Waxes (D 1387 - 55 T)

Committee D-23 on Cellulose and Cellulose Derivatives:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Accepted as Tentative:

Test for Solubility of Cellulose in Sodium Hydroxide (D 1696 - 59 T)
Def. of Terms Relating to Cellulose and Cellulose Derivatives (D 1695 - 59 T)

Committee D-26 on Halogenated Organic Solvents:

Report presented in the absence of the chairman by W. D. McMaster, and accepted as a report of progress.

TWENTY-NINTH SESSION—SYMPOSIUM ON MICROSCOPY[18]

THURSDAY, JUNE 25, 2:00 P.M.

SESSION CHAIRMAN: F. F. MOREHEAD

Introduction—F. F. Morehead, presented by the author.

Measurement with Phase and Interference Microscopes—O. W. Richards, presented by the author.

Performance Characteristics of Objectives, Eyepieces and Illuminators for Microscopy (Informal Presentation)—H. W. Zieler, presented by the author.

Chemical Microscopy in the Optical Industry—G. C. Crossmon, presented by the author.

Study of Opaque Minerals in Reflected Light—E. N. Cameron, presented by the author.

(Continued in the Thirty-Second and Thirty-Fourth Sessions)

---

[18] Issued as separate publication *ASTM STP No. 257.*

THIRTIETH SESSION—SYMPOSIUM ON VISUAL AIDS FOR STANDARDIZING AND COMMUNICATING PRODUCT APPEARANCE[19]

THURSDAY, JUNE 25, 2:00 P.M.

SESSION CHAIRMAN: J. M. HEMPHILL

Welcome and Introduction—J. M. Hemphill, presented by the author.

Status of ASTM Methods and Standards for Appearance Evaluation—I. Nimeroff, presented by the author.

Visual Aids in the Textile Industry—J. B. Goldberg, presented by the author.

Potential Uses of Closed Circuit Television for Product Inspection—R. Vendeland, presented by the author.

Use of Color Photography for Product Specification and Control—J. R. Kane, presented by the author.

Summation—G. W. Ingle, presented by the author.

---

[19] Issued as separate publication *ASTM STP No. 258.*

NUCLEAR PROBLEMS FORUM

THURSDAY, JUNE 25, 2:00 P.M.

Sponsored by Special Administrative Committee on Nuclear Problems

Sponsored by the ASTM Special Administrative Committee on Nuclear Problems, this forum was intended to familiarize the people engaged in nuclear energy projects with the work of the technical committees. In this manner it is hoped to exchange information on standardization work being done in the ASTM committees and on work which should be considered.

THIRTY-FIRST SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 25, 4:30 P.M.

SESSION CHAIRMAN: A. C. WEBBER

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by R. A. Harris, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum Alloy Pipe for Gas and Oil Transmission and Distribution Piping Systems (B 345 - 59 T)

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Base Alloy Sand Castings (B 26 - 58 T)
Spec. for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 58 T)
Spec. for Aluminum-Alloy Sheet and Plate (B 209 - 58 T)
Spec. for Aluminum-Alloy Drawn Seamless Tubes (B 210 - 58 T)
Spec. for Aluminum-Alloy Bars, Rods, and Wire (B 211 - 58 T)
Spec. for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 58 T)
Spec. for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 58 T)
Spec. for Aluminum-Alloy Extruded Tubes (B 235 - 58 T)
Spec. for Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 - 58 T)
Spec. for Aluminum-Alloy Pipe (B 241 - 58 T)
Spec. for Aluminum-Alloy Die Forgings (B 247 - 58 T)
Spec. for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 - 58 T)
Spec. for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 - 58 T)
Spec. for Aluminum-Alloy Round Welded Tubes (B 313 - 58 T)
Spec. for Aluminum-Alloy Rivet and Cold Heading Wire and Rods (B 316 - 58 T)
Spec. for Extruded Aluminum-Alloy Bars, Rods, Pipe, and Structural Shapes for Electrical Purposes (Bus Conductors) (B 317 - 58 T)
Spec. for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318 - 58 T)

Adopted as Standard, Revisions in:

Spec. for Aluminum for Use in Iron and Steel Manufacture (B 37 - 57)
Spec. for Magnesium-Base Alloy Sand Castings (B 80 - 58)
Spec. for Magnesium-Base Alloy Sheet and Plate (B 90 - 58)
Spec. for Magnesium-Base Alloy Forgings (B 91 - 58)
Spec. for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 93 - 58)
Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 - 58)
Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 58)
Spec. for Magnesium-Base Alloy Permanent Mold Castings (B 199 - 58)
Spec. for Magnesium-Base Alloy Extruded Tubes (B 217 - 58)
Rec. Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 - 58)

Committee C-17 on Asbestos-Cement Products:

Report presented by W. V. Friedlaender, chairman, and accepted as a report of progress.

Committee D-5 on Coal and Coke:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and accepted as a report of progress.

Committee D-17 on Naval Stores:

Report presented by S. R. Snider, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Tests for Saponification Number of Rosin (D 464 - 51)
Tests for Acid Number of Rosin (D 465 - 51)

Committee D-20 on Plastics:

Report presented by F. W. Reinhart, chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Exposure of Polymeric Materials to High Energy Radiation (D 1672 - 59 T) (Joint with Committee D-9)
Test for Gamma Radiation by Chemical Dosimetry (D 1671 - 59 T) (Joint with Committee D-9)

Test for Flammability of Plastics Foams and Sheeting (B 1692 - 59 T), revised by the deletion of Section 6(*c*), and the addition of the following new paragraph (*4*) to Section 8 on Report, renumbering the present paragraph (*4*) and subsequent paragraphs accordingly:

(*4*) Classification of the material as "nonburning by this test," "self-extinguishing by this test," or "burning by this test."

Test for Environmental Stress-Cracking of Type I Ethylene Plastics (D 1693 - 59 T), revised by the deletion in Section 10 of "which shall be 48 hr unless otherwise stated," and the addition of the sentence "Not more than 5 shall fail." It was reported that a revision is in process of Section 7 on Preparation of Test Specimen.

Spec. for Thermosetting Reinforced Plastic Pipe Thread System (D 1694 - 59 T)

Accepted as Tentative, Revisions in:

Test for Water Absorption of Plastics (D 570 - 57 T), with the deletion of the revision of Section 6(*a*).

Test for Tear Resistance of Plastic Film and Sheeting (D 1004 - 49 T)

Test for Dilute Solution Viscosity of Ethylene Polymers (D 1601 - 58 T), with the following change in Section 8(*f*):

(*f*) Remove the lower tip of about 0.5 in. from a transfer pipet to permit faster transfer of its contents to the viscometer. Place the pipet in a 140 C oven for 30 min. It is preferable that the pipet be fitted with a suitable beating mantle to retard precipitation of the polymer on its walls during transfer of the solution (Note 11). Purge the viscometer thoroughly with nitrogen gas using a slow, gentle stream. Fill the pipet from the sample solution flask and transfer approximately 15 ml of the solution into the filter stick of the modified Ubbelohde viscometer which is permanently positioned in the bath (Note 12). (See the Appendix to this method for directions for use and cleaning of the viscometer.)

Add the following new Notes 11 and 12; renumber Note 10 as Note 13, and the present Note 11 as Note 14:

NOTE 11.—A graduate cylinder previously heated in the viscosity bath may be used instead of a pipet. The hot solution can be poured into the graduate positioned in the bath, the graduate removed after about 3 min and its contents poured into the viscometer filter stick.

NOTE 12.—If the dilution method described in Note 8 is used, exactly 15 ml of solution must be transferred to the viscometer using the transfer pipet described above.

Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 - 58) (Joint with Committee D-9)

Adopted as Standard, Revisions in:

Test for Measuring the Flow Properties of Thermoplastic Molding Materials (D 569 - 48)

Tests for Deformation of Plastics Under Load (D 621 - 51)

Standard Withdrawn:

Spec. for Cast Allyl Plastic Sheets, Rods, Tubes, and Shapes (D 819 - 50)

Committee D-20 withdrew from the report as preprinted the recommendations for adoption as standard of Tentative Methods of Test D 1494 - 57 T, D 1502 - 57 T, and D 1504 - 57 T, and the reapproval of Standard Method of Test D 793 - 49.

Committee D-24 on Carbon Black:

Report presented by N. P. Bekema, chairman, and the following actions taken:

Adopted as Standard:

Test for Ash Content of Carbon Black (D 1506 - 57 T)

Test for Heating Loss of Carbon Black (D 1509 - 57 T)

Committee E-1 on Methods of Testing:

Report presented by P. J. Smith, secretary, and the following actions taken:

Accepted as Tentative:

Specs. for Gravity-Convection and Forced-Ventilation Laboratory Ovens (E 145 - 59 T)

Method for Determination of Shear Modulus at Room Temperature (E 143 - 59 T)

Rec. Practice for Safe Use of Oxygen Combustion Bombs (E 144 - 59 T)

Accepted as Tentative, Revisions in:

Method for Determination of Young's Modulus at Room Temperature (E 111 - 58 T)

Spec. for ASTM Hydrometers (E 100 - 58T)

Method for Inspection and Verification of Hydrometers (E 126 - 57 T)

Spec. for ASTM Thermometers (E 1 - 58)

Adopted as Standard, Revisions in:

Spec. for ASTM Thermometers (E 1 - 58)

Committee E-7 on Nondestructive Testing:

Report presented by J. H. Bly, chairman, and the following action taken:

Accepted as Tentative:

Method for Controlling Quality of Radiographic Testing (E 142 - 59 T)

Committee E-12 on Appearance:

Report presented by G. W. Ingle, chairman, and accepted as a report of progress.

Joint Committee on Leather:

Report presented in the absence of the chairman by B. L. Lewis, vice-chairman, and the following action taken:

Accepted as Tentative:

Test for Bond Strength of Leather Belting (D 1699 - 59 T)

THIRTY-SECOND SESSION—SYMPOSIUM ON MICROSCOPY[18]

(Continued from the Twenty-Ninth Session)

THURSDAY, JUNE 25, 8:00 P.M.

SESSION CHAIRMAN: M. C. MILLER

Application of Electron Microscopy in the Petroleum Industry—H. Allred, presented by the author.

The Electron Microscope in the Study of Minerals and Ceramics—J. E. Comer, presented by the author.

Interpretation of Light and Electron Microscopical Observations—C. F. Tufts, presented by the author.

(Continued in the Thirty-Fourth Session)

THIRTY-THIRD SESSION—SYMPOSIUM ON IDENTIFICATION OF WATER-FORMED DEPOSITS[20]

THURSDAY, JUNE 25, 8:00 P.M.

SESSION CHAIRMAN: R. K. SCOTT

Presentation of Max Hecht Award:

The Sixth Max Hecht Award was given to L. Drew Betz, General Manager, Betz Laboratories, Inc., Philadelphia, Pa., who has performed outstanding work in Committee D-19 and in the field of industrial water.

Committee D-19 on Industrial Water:

Report presented by Max Hecht, chairman, and the following actions taken:

Accepted for Publication as Information Only:

Procedure for Determination of Precision of Committee D-19 Methods.

Accepted as Tentative:

Test for Copper in High-Purity Water (D 1688 - 59 T)

Test for Silica in High-Purity Water (D 1689 - 59 T)

Method for Measurement of Gamma Radioactivity of Industrial Water and Industrial Waste Water (D 1690 - 59 T)

Test for Total Chromium in Industrial Water and Industrial Waste Water (D 1687 - 59 T)

Test for Zinc in Industrial Water and Industrial Waste Water (D 1691 - 59 T)

Accepted as Tentative, Revisions in:

Tests for Sulfate Ion in Industrial Water and Industrial Waste Water (D 516 - 55 T)

Method of Sampling Steam (D 1066 - 54 T)

Test for Electrical Conductivity of Industrial

---

[20] Issued as separate publication *ASTM STP No. 256.*

Water and Industrial Waste Water (D 1125 - 50 T)
Scheme for Analysis of Industrial Water and Industrial Waste Water (D 1256 - 53 T)

Adopted as Standard:

Test for Oxidation-Reduction Potential of Industrial Water (D 1498 - 57 T)
Test for Sulfides in Industrial Waste Water (D 1255 - 55 T)
Test for Evaluating Acute Toxicity of Industrial Waste Water to Fresh-Water Fishes (D 1345 - 54 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 - 58)

Symposium Papers:

Deposit Identification—The First Step Toward Understanding a Water Problem—J. K. Rice, presented by the author.
Application of Emission Spectroscopy to the Analysis of Water-Formed Deposits—C. H. Anderson, presented by the author.
Identification by Instrumental Methods of Chemical Compounds in Water-Formed Deposits—C. M. Maddin and R. B. Rosene, presented by Mr. Maddin.
Electron Microscopy and Electron Diffraction Studies of Oxide Films Formed on Iron in Water Vapor and Oxygen Atmospheres—E. A. Gulbransen and T. P. Copan, presented by Mr. Copan.
Correlation of Elemental Analysis and Phase Identification as Viewed by a Mineralogist—J. V. Smith, presented by the author.
Summary—R. K. Scott, presented by the author.

STATISTICAL TREATMENT OF INTERLABORATORY TEST RESULTS, INCLUDING A NEW GRAPHICAL METHOD

THURSDAY, JUNE 25, 8:00 P.M.

Sponsored by Committee C-1 on Cement

A new graphic method for statistical treatment of interlaboratory test results was described. The method affords a concise means of measuring the effectiveness of test procedures or specification methods and a means for evaluating the degree of skill exhibited by different laboratories. Application of this technique to a Cement Reference Sample Program consisting of interlaboratory tests on 12 cements by 103 laboratories was described and discussed by Messrs. Blaine, Crandall, and Youden of the National Bureau of Standards.

The Cement Reference Sample Program, Introduction—R. L. Blaine.
A New Graphic Method for Statistical Treatment and Evaluation of Interlaboratory Tests—W. J. Youden.
Application of the Youden Technique to Cement Tests—J. R. Crandall.
Discussion of the Significance of the Results of the Reference Sample Program to Cement Testing—R. L. Blaine.
Application of the Graphic Method to Other Interlaboratory Testing and Specifications—W. J. Youden.

THIRTY-FOURTH SESSION—SYMPOSIUM ON MICROSCOPY[18]

(Continued from the Twenty-Ninth and Thirty-Second Sessions)

FRIDAY, JUNE 26, 9:30 A.M.

SESSION CHAIRMAN: L. L. WYMAN

The Structure of Synthetic Fibers—R. G. Scott, presented by the author.
Microscopical Studies of Failure in Polymers—S. B. Newman, presented by Mr. Weissberg.
Microscopical Evaluation of Cotton Finishing Treatment—M. L. Rollins, I. V. DeGruy, V. M. Tripp, and A. T. Moore, presented by Miss Rollins.

THIRTY-FIFTH SESSION—COMMITTEE REPORT SESSION

FRIDAY, JUNE 26, 12:30 P.M.

SESSION CHAIRMAN: M. N. CLAIR

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts:

Report presented by E. I. Shobert, II, chairman, and the following actions taken:

Accepted as Tentative:

Method of Accelerated Life Test of Iron-Chromium-Aluminum Alloys for Electrical Heating (B 78 - 59 T)

Adopted as Standard:

Specs. for Drawn or Rolled Nickel-Chromium and Nickel-Chromium-Iron Alloys for Electrical Heating Elements (B 344 - 59) (New Standard)

Adopted as Standard, Revisions in:

Method of Accelerated Life Test for Metallic Materials for Electrical Heating (B 76 - 57)

Standards Withdrawn:

Spec. for Drawn or Rolled Alloy, 80 Per Cent Nickel, 20 Per Cent Chromium, for Electrical-Heating Elements (B 82 - 57)
Spec. for Drawn or Rolled Alloy, 60 Per Cent Nickel, 16 Per Cent Chromium, and Balance Iron, for Electrical-Heating Elements (B 83 - 52)

Committee C-1 on Cement:

Report presented in the absence of the chairman by M. N. Clair, and the following actions taken:

Accepted as Tentative:

Test for Fineness of Hydraulic Cement by the No. 325 Sieve (C 430 - 59 T)

Accepted as Tentative, Revisions in:

Method for Mechanical Mixing of Hydraulic Cement Mortars of Plastic Consistency (C 305 - 58 T)
Specs. for Portland Cement (C 150 - 56)
Specs. for Air-Entraining Portland Cement (C 175 - 56)
Test for Fineness of Portland Cement by Air Permeability Apparatus (C 204 - 55)

Adopted as Standard:

Test for Air Content of Hydraulic Cement Mortar (C 185 - 58 T)

Adopted as Standard, Revisions in:

Spec. for Masonry Cement (C 91 - 58)
Spec. for Portland Cement (C 150 - 56)
Test for Autoclave Expansion of Portland Cement (C 151 - 58)
Spec. for Air-Entraining Portland Cement (C 175 - 56)
Test for Tensile Strength of Hydraulic Cement Mortars (C 190 - 58)

Committee C-11 on Gypsum:

Report presented in the absence of the chairman by R. H. Faber, secretary, and the following actions taken:

Accepted as Tentative:

Specs. for Gypsum Backing Board (C 442 - 59 T)

Adopted as Standard:

Spec. for Inorganic Aggregates for Use in Gypsum Plaster (C 35 - 57 T)

Adopted as Standard, Revisions in:

Methods of Testing Gypsum and Gypsum Products (C 26 - 56)
Spec. for Gypsum Plasters (C 28 - 58)
Def. of Terms Relating to Gypsum (C 11 - 58)

Committee C-15 on Manufactured Masonry Units:

Report presented by J. W. Whittemore, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126 - 58 T)
Spec. for Clay Drain Tile (C 4 - 55) (Standard Reverted to Tentative)

This recommendation had been indicated in the preprinted report as a new tentative.

Adopted as Standard, Revisions in:

Spec. for Hollow Load-Bearing Concrete Masonry Units (C 90 – 52)
Spec. for Hollow Non-Load-Bearing Concrete Masonry Units (C 129 – 52)
Spec. for Solid Load-Bearing Concrete Masonry Units (C 145 – 52)

Committee E-2 on Emission Spectroscopy:

Report presented by D. L. Fry, chairman, and the following actions taken:

Accepted for Publication as Information Only:

Method for Spectrochemical Analysis of Antimony by the Point-to-Plane Spark Technique
Method for Spectrochemical Analysis of Magnesium-Base Alloys by the Point-to-Plane Spark Technique Using a Grating Spectrograph

Accepted as Tentative, Revisions in:

Method of Spectrochemical Analysis of Zinc-Base Alloys and High-Grade Zinc by the Solution – D-C Arc Technique (E 27 – 53 T)
Method for Spectrochemical Analysis of Tin Alloys for Minor Constituents and Impurities (E 51 – 43 T)
Method for Spectrochemical Analysis of Pig Lead by the Point-to-Plane Spark Technique (E 117 – 56 T)

The committee withdrew from the report as preprinted the recommendation for publication as information of the Method for Spectrochemical Analysis of Zinc-Base Die-Casting Alloys by the Point-to-Plane Spark and A-C Arc Techniques.

Committee E-13 on Absorption Spectroscopy:

Report presented in the absence of the chairman by R. F. Robey, secretary, and the following actions taken:

Accepted for Publication as Information Only:

Rec. Practice for Form of ASTM Methods Relating to Absorption Spectroscopy
Rec. Practice for General Techniques of Infrared Quantitative Analysis
Rec. Practice for General Techniques of Ultraviolet Quantitative Analysis

Accepted as Tentative, Revisions in:

Def. of Terms and Symbols Relating to Absorption Spectroscopy (E 131 – 57 T)

THIRTY-SIXTH SESSION—COMMITTEE REPORT SESSION

FRIDAY, JUNE 26, 12:30 P.M.

SESSION CHAIRMAN: R. C. ALDEN

Committee D-2 on Petroleum Products and Lubricants:

Report presented in the absence of the chairman by W. T. Gunn, secretary, and the following actions taken:

Accepted for Publication as Information Only:

Proposed Method of Test for Smoke Density in the Flue Gases from Distillate Fuels
Proposed Method of Test for Motor and Research Octane Numbers Permitting the Use of Small Samples
Proposed Method of Test for Naphthalene in Aviation Turbine Fuels by Ultraviolet Spectroscopy
Proposed Definitions of Fluid Bulk Modulus
Cooperative Data Showing Comparison of Results Using Apparatus Specified in Tentative Method D 1319 and "Chromanalyzer" Apparatus
Proposed Method of Test for Filterability of Aviation Turbine Fuels
Proposed Method for Humidity Cabinet Corrosion Tests
Proposed Method of Test for Luminosity Numbers of Aviation Turbine Fuels
Proposed Method of Test for Particulate Matter in Hydrocarbons
Report on Stable Pour Point by the Cycle C Method

The last four proposed items were not referred to in the preprinted report but were presented at the Session.

Accepted as Tentative:

Test for Maximum Fluidity Temperature of Residual Fuel Oil (D 1659 – 59 T)
Test for Thermal Stability of Aviation Turbine Fuels (D 1660 – 59 T)
Test for Knock Characteristics of Motor Fuels Above 100 Octane Number by the Research Method (D 1656 – 59 T)

Test for Specific Gravity of Light Hydrocarbons by Pressure Hydrometer (D 1657 - 59 T)
Test for Carbon Number Distribution of Aromatic Compounds in Naphthas by Mass Spectrometry (D 1658 - 59 T)
Test for Thermal Stability of Navy Special Fuel Oil (D 1661 - 59 T)
Test for Active Sulfur in Cutting Fluids (D 1662 - 59 T)
Spec. for Aviation Turbine Fuels (D 1655 - 59 T)

Accepted as Tentative, Revisions in:

Test for Water and Sediment by Means of Centrifuge (D 96 - 58 T)
Test for Distillation of Plant Spray Oils (D 447 - 57 T)
Test for Ignition Quality of Diesel Fuels by the Cetane Method (D 613 - 58 T)
Test for Knock Characteristics of Aviation Fuels by the Aviation Method (D 614 - 58 T)
Test for Sulfated Residue from New Lubricating Oils (D 874 - 57 T)
Test for Knock Characteristics of Aviation Fuels by the Supercharge Method (D 909 - 58 T)
Classification of Diesel Fuel Oils (D 975 - 53 T)
Test for Peroxides in Butadiene (Ferrous-Titanous Method) (D 1022 - 49 T)
Test for Bromine Number of Petroleum Distillates (Electrometric Method) (D 1159 - 57 T)
Methods for Calibrating Upright Tanks (D 1220 - 58 T)
Test for Sulfur in Petroleum Products Including Liquefied Petroleum Gas by Lamp Combustion (D 1266 - 57 T)
Tests for Sodium in Residual Fuel Oils by Flame Photometer (D 1318 - 54 T)
Test for Smoke Point of Jet Fuels (D 1322 - 58 T)
Test for Vanadium in Navy Special Fuel Oil (D 1548 - 58 T)
Test for Ash Content of Petroleum Oils (D 482 - 46) (Standard Reverted to Tentative)

Adopted as Standard:

Test for Carbon Residue of Petroleum Products (Ramsbottom Coking Method) (D 524 - 58 T)

Adopted as Standard, Revisions in:

Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 56)
Methods of Analysis of Lubricating Grease (D 128 - 57)
Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158 - 54)
Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357 - 58)
Test for Sediment in Fuel Oil by Extraction (D 473 - 48)
Tests for Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils (D 810 - 48)
Test for Knock Characteristics of Motor Fuels by the Research Method (D 908 - 58)
Test for Butadiene Content of Polymerization Grade Butadiene (D 973 - 50)
Test for Acetylene in Polymerization Grade Butadiene (Silver Nitrate Method) (D 1020 - 52)
Test for Butadiene Dimer in Polymerization Grade Butadiene (D 1024 - 53)
Test for Carbonyl Content of Butadiene (D 1089 - 53)
Test for Total Inhibitor Content (*p*-Tertiary-Butyl-Catechol) of Butadiene (D 1157 - 54)

The committee withdrew from the report as preprinted the recommendations for revision and reversion to tentative of the Standard Method of Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 - 58), and the continuation without revision of the Tentative Method of Test for Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus (D 1310 - 56 T).

The change in Tentative Method D 1158 - 57 T is editorial in nature but was inadvertently indicated in the Preprinted Report as a substance change.

Committee D-4 on Road and Paving Materials:

Report presented by A. B. Cornthwaite, chairman, and the following actions taken:

Accepted as Tentative:

Test for Engler Specific Viscosity of Tar Products (D 1665 - 59 T)
Test for Coating and Stripping of Bitumen-Aggregate Mixtures (D 1664 - 59 T)
Test for Penetration of Bituminous Materials (D 5 - 59 T)
Specs. for Hot-Mixed, Hot-Laid Asphalt Paving Mixtures (D 1663 - 59 T)

Accepted as Tentative, Revisions in:

Spec. for Crushed Stone, Crushed Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 – 54) (Standard Reverted to Tentative)
Spec. for Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 – 54) (Standard Reverted to Tentative)

Adopted as Standard:

Spec. for Calcium Chloride (D 98 – 56 T)
Method of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 – 58 T)
Test for Scratch Hardness of Coarse Aggregate Particles (C 235 – 57 T)

Method C 235 is under joint jurisdiction of Committees D-4 and C-9; the recommendation for adoption as standard failed of approval in Committee C-9 and has not been included in the letter ballot of the Society.

Adopted as Standard, Revisions in:

Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 42) (Joint with Committee C-9)
Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 57) (Joint with Committee C-9)

The committee withdrew from the report as preprinted the recommendations for the withdrawal of Standard Specifications D 947 – 55 and D 978 – 54.

Committee D-6 on Paper and Paper Products:

Report presented by R. H. Carter, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Puncture and Stiffness of Paperboard, Corrugated, and Solid Fiberboard (D 781 – 44 T)

Adopted as Standard, Revisions in:

Method of Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 – 48)
Tests for Ply Separation of Combined Container Board (D 1028 – 51)
Test for Peeling Resistance of Paperboard (D 1029 – 52)

Tentative Withdrawn:

Test for Dimensional Changes of Paper With Changes in Moisture Conditions (D 1270 – 53 T)

The committee withdrew from the report as preprinted the recommendations for publication as tentative of the Method of Test for Flat Crush of Corrugating Medium (CMT), and the revision for immediate adoption of the Standard Method of Test for Absorption by Bibulous Papers of Water and Writing Ink (D 824 – 47).

Committee D-9 on Electrical Insulating Materials:

Report presented by H. K. Graves, chairman, and the following actions taken:

Accepted for Publication as Information Only:

Method for Evaluation of Thermal Stability of Electrical Insulating Coated Sleevings (Dielectric Breakdown Method)

Accepted as Tentative:

Test for Gamma Radiation by Chemical Dosimetry (D 1671 – 59 T) (Joint with Committee D-20)
Rec. Practice for Exposure of Polymeric Materials to High Energy Radiation (D 1672 – 59 T) (Joint with Committee D-20)
Tests for Dielectric Constant and Dissipation Factor of Expanded Cellular Plastics Used for Electrical Insulation (D 1673 – 59 T)
Methods of Testing Electrical Grade Polytetrafluoroethylene Tubing (D 1675 – 59 T)
Methods of Sampling and Testing Untreated Mica Paper Used for Electrical Insulation (D 1677 – 59 T)
Tests for Frictional Characteristics of Enameled Magnet Wire for Use in Winding Filled Coils (D 1676 – 59 T)

Accepted as Tentative, Revisions in:

Spec. for Orange Shellac and Other Indian Lacs for Electrical Insulation (D 784 – 52 T)
Methods of Testing Pressure-Sensitive Adhesive Coated Tapes Used for Electrical Insulation (D 1000 – 58 T)
Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 58) (Joint with Committee D-20)

Adopted as Standard:

Spec. for Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors (D 748 – 54 T)
Spec. for Nonrigid Polyvinyl Tubing (D 922 – 54 T)
Spec. for Silicone Varnished Glass Cloth and Tape for Electrical Insulation (D 1459 – 57 T)
Test for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating

Materials at Commercial Power Frequencies (D 149 – 59 T)
Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 – 56 T)
Methods of Testing Pasted Mica Used in Electrical Insulation (D 352 – 56 T)
Test for Dissipation Factor and Dielectric Constant Parallel with Laminations of Laminated Sheet and Plate Insulating Materials (D 669 – 42 T)
Rec. Practice for Cleaning Plastic Specimens for Insulation Resistance Testing (D 1371 – 55 T)

The committee withdrew from the report as preprinted the recommendations for publication as tentative of the Methods of Test for Heat Resistance and Thermal Stability of Rigid Electrical Insulation, and the Definitions of Terms Relating to Electrical Insulating Materials, and the adoption as standard of Tentative Specifications for Natural Muscovite Mica Based on Visual Quality (D 351 – 57 T).

Committee D-11 on Rubber and Rubber-like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Synthetic Rubber Heat- and Moisture-Resisting Insulation for Wire and Cable, 75 C Operation (D 1679 – 59 T)
Methods of Testing Automotive Air Conditioning Hose (D 1680 – 59 T)

Accepted as Tentative, Revisions in:

Methods of Sample Preparation for Physical Testing of Rubber Products (D 15 – 58 T)
Spec. for Friction Tape for General Use for Electrical Purposes (D 69 – 57 T)
Spec. for Rubber Insulating Tape (D 119 – 57 T)
Methods of Testing Rubber and Thermoplastic Insulated Wire and Cable (D 470 – 58 T)
Test for Change in Properties of Elastomeric Vulcanizates Resulting from Immersion in Liquids (D 471 – 57 T)
Spec. for Elastomer Compounds for Automotive Applications (D 735 – 58 T), with the addition of the following note to appear after the revised Section 5(*h*):

> NOTE.—Some upgrading of quality may be required in some cases because of the slightly greater severity of Method D 746 over the method previously specified, namely, Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (ASTM Designation: D 736).

Test for Compressibility and Recovery of Gasket Materials (D 1147 – 56 T)
Spec. for Nonmetallic Gasket Materials for General Automotive and Aeronautical Purposes (D 1170 – 58 T)
Spec. for Ozone Resistant Rubber Insulating Tape (D 1373 – 57 T)
Rec. Practice for Description of Types of Styrene-Butadiene Rubbers (SBR) (D 1419 – 59 T), changing in Table II the Mooney viscosity value for Type 1507 from "40" to "35."
Rec. Practice for Description of Types of Styrene-Butadiene Rubber (SBR) and Butadiene Rubber (BR) Latices (D 1420 – 59 T), with the further addition to Table I of type 2109 with the following properties:

	TYPE 2109
Nominal Temperature, deg Fahr	50
Activator	FRA
Shortstop	ND
Catalyst	OHP
Emulsifier	FA
Nominal Conversion, per cent	60
Contained Polymer Nominal Mooney Viscosity, ML 1 + 4 (212 F)	...
Nominal Residual Volatile Unsaturate, per cent	0.1
Nominal pH Value	10.5
Nominal Surface Tension, dynes per cm	...
Nominal Coagulum on No. 80 Screen, per cent	0.10
Nominal Bound Styrene, per cent	40
Nominal Total Solids, per cent	39.5

Adopted as Standard:

Spec. for Rubber Insulating Blankets (Without Fabric Reinforcement) (D 1048 – 49 T)
Spec. for Rubber Insulator Hoods (D 1049 – 49 T)
Spec. for Rubber Insulating Line Hose (D 1050 – 49 T)
Spec. for Rubber Insulating Sleeves (D 1051 – 56 T), with the title of Section 3(*a*) changed to read: "Voltage Test and Proof Test Current"; also Section 6 revised to read: "The sleeves shall be free from harmful physical defects which can be detected by thorough test or inspection."
Spec. for Concentrated, Ammonia Preserved, Creamed and Centrifuged Natural Rubber Latex (D 1076 – 57 T)
Spec. for Nonrigid Thermoplastic Compounds for Automotive and Aeronautical Applications (D 1277 – 53 T)
Spec. for Sheet Rubber Packing (D 1330 – 55 T)
Methods of Testing Rubber Hose (D 380 – 57 T)
Methods of Dynamic Testing for Ply Separa-

tion and Cracking of Rubber Products (D 430 – 57 T)
Methods of Testing Compressed Asbestos Sheet Packing (D 733 – 57 T)
Test for Resistance of Vulcanized Rubber or Synthetic Elastomers to Crack Growth (D 813 – 57 T)
Rec. Practice for Conditioning of Elastomeric Materials for Low-Temperature Testing (D 832 – 56 T)
Test for Weather Resistance Exposure of Automotive Rubber Compounds (D 1171 – 57 T)
Test for Penetration of Hard Rubber by Type D Durometer (D 1484 – 57 T)
Methods for Testing Synthetic Rubber Latices (Styrene-Butadiene Copolymers) (D 1417 – 57 T), with the further addition to Table I of latices 2076, 2107, 2108, 2109, 2110, 2111 and 2112, with a drying aid of 1 ml of distilled water and a drying time of 45 min.

Adopted as Standard, Revisions in:

Test for Abrasion Resistance of Rubber Compounds (D 394 – 47)
Tests for Mechanical Properties of Elastomeric Vulcanizates Under Compressive or Shear Strains by the Mechanical Oscillograph (D 945 – 55)

The committee withdrew from the report as preprinted the recommendation for adoption as standard with revision of the Tentative Specifications for Rubber Insulating Gloves (D 120 – 52 T). The revision in question will be submitted at a later date to the Society through the Administrative Committee on Standards.

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials:

Report presented by W. E. Sisco, chairman, and the following actions taken:

Accepted for Publication as Information Only:

Test for *o*-Xylene, *m*-Xylene, *p*-Xylene, and Ethylbenzene in Nitration Grade Xylene by Infrared Spectrophotometry

Accepted as Tentative:

Test for Color of Solid Aromatic Hydrocarbons and Related Materials in the Molten State (Platinum-Cobalt Scale) (D 1686 – 59 T), with the addition of the following Section 7(*c*):

(*c*) If, owing to large differences in hue between the sample and the standards no estimate is possible, report the sample as "no match."

Test for Traces of Thiophene in Benzene Using Isatin and Spectrophotometry (D 1685 – 59 T)

Adopted as Standard, Revisions in:

Test for Specific Gravity of Industrial Aromatic Hydrocarbons (D 891 – 51)

Committee E-11 on Quality Control of Materials:

Report presented by Simon Collier, chairman, and accepted as a report of progress.

EDITOR'S NOTE.—The recommendations affecting standards referred to letter ballot were approved when the ballot was canvassed on September 10.

SUMMARY OF PROCEEDINGS OF THE THIRD PACIFIC AREA NATIONAL MEETING

San Francisco, Calif., October 11–16, 1959

The Third Pacific Area National Meeting of the Society was held at the Sheraton-Palace Hotel in San Francisco on October 11–16, 1959, with the Northern and Southern California ASTM District Councils cooperating in making arrangements for the meeting. The officers of the General Committee on Arrangements are:

Chairman, P. V. Garin, Southern Pacific Co.
Vice-Chairman, P. E. McCoy, American Bitumuls & Asphalt Co.,
Vice-Chairman, M. B. Niesley, California Testing Labs., Inc.,
Secretary, H. P. Hoopes, Fibreboard Paper Products Co., and
Treasurer, L. A. O'Leary, W. P. Fuller & Co.

Subcommittee Chairmen:

Finance

Chairman, L. A. O'Leary, W. P. Fuller & Co.
Vice-Chairman, Jonmore Dickason, Metal Control Labs., Inc.
Vice-Chairman, E. W. Gardiner, California Research Corp.

Technical Program

Chairman, G. J. Grieve, Pacific Paint & Varnish Co.
Vice-Chairman, R. C. Vollmar, Standard Oil Co. of Calif.
Vice-Chairman, Ernst Maag, Calif. State Dept. of Public Wks.

Social

Chairman, R. W. Harrington, Clay Brick & Tile Assn.
Vice-Chairman, H. de Bussieres, Curtis & Tompkins, Ltd.

Transportation

Chairman, C. F. Lapier, Matson Navigation Co.
Vice-Chairman, R. A. Kinzie, Jr., Pacific Cement & Aggregates, Inc.

Hotels

Chairman, W. W. Moore, Dames & Moore
Vice-Chairman, R. C. Kennedy, East Bay Municipal Util. Dist.

Information

Chairman, C. F. Fitzwilson, Columbia-Geneva Steel Div.
Vice-Chairman, G. F. Scherer, Rockwell Mfg. Co.

Promotion and Publicity

Chairman, T. K. Cleveland, Phila. Quartz Co. of Calif.
Vice-Chairman, R. N. Connor, Baldwin-Lima Hamilton Corp.
Vice-Chairman, F. W. Twining, Twining Laboratories

Plant Visits

Chairman, E. V. Noe, Pacific Gas & Elec. Co.
Vice-Chairman, M. C. Poulsen, Port Costa Brick Works

Industry Luncheons

Chairman, Roy Henning, Eitel-McCullough, Inc.
Vice-Chairman, Don Bowers, General Petroleum Corp.

The registered attendance at the meeting was as follows: 1302 members, committee members, and visitors, plus 149 ladies, or a grand total of 1451.

There were 52 technical sessions with 222 papers scheduled for presentation. Many of these sessions were sponsored by technical committees of the Society.

In addition, there were five industry luncheons held throughout the week, with a "President's Luncheon" on Wednesday, October 14. The technical sessions and the luncheons are set forth below.

PRESIDENT'S NATIONAL AND INDUSTRY LUNCHEONS

The President's National Luncheon and a number of special "Industry Luncheons" were arranged to provide an opportunity for gatherings of those interested in special fields. These luncheons, together with the listing of the toastmasters and guest speakers, are given below.

Statistics Luncheon MONDAY, OCTOBER 12, 12 NOON

Toastmaster: Past President Richard T. Kropf, Vice-President and Director of Research, Belding Heminway Co., Inc., New York.

Guest Speaker: Frank Squires, Assistant to the President, Topp Industries, Inc., Los Angeles

Subject: "From Certainty to Statistics"[1]

Collaborating Committee: Committee E-11 on Quality Control of Materials

Electronics Industry Luncheon TUESDAY, OCTOBER 13, 12 NOON

Toastmaster: Past President R. A. Schatzel, Vice-President and Director of Engineering, Rome Cable Corp., Rome, N. Y.

Guest Speaker: J. A. Chartz, President, Western Electronic Mfg. Assn., Dalmo Victor Co., Belmont, Calif.

Subject: "Our Products *Versus* Your Tests"[2]

Collaborating Committees: Committees A-6 on Magnetic Properties, C-21 on Ceramic Whitewares and Related Properties, D-9 on Electrical Insulating Materials, F-1 on Materials for Electron Tubes and Semiconductor Devices

Petroleum and Chemical Industry Luncheon TUESDAY, OCTOBER 13, 12 NOON

Toastmaster: Past National Director R. C. Alden, Consultant, Phillips Petroleum Co.

Guest Speaker: T. L. Lenzen, Director and Vice-President, Standard Oil Company of California, San Francisco

Subject: "Foreign Petroleum"[2]

Collaborating Committee: Committee D-2 on Petroleum Products and Lubricants

---

[1] Published in the ASTM BULLETIN, No. 243, Jan., 1960, p. 27.

[2] To be published in the ASTM BULLETIN.

President's National Luncheon WEDNESDAY, OCTOBER 14, 12 NOON

In honor of President F. L. LaQue, Vice-President and Manager, Development and Research Division, The International Nickel Co., Inc., New York.

As a special guest of honor, Lt. Gen. Bernard A. Schriever, Commander, Air Research and Development Command, U. S. Air Force, will deliver the principal address.

Toastmaster: Past National Director Paul V. Garin, Manager, Research and Development, Southern Pacific Co., San Francisco.

Short Address by President LaQue[3]
Main Address by General Schriever[4]

Water Industry Luncheon THURSDAY, OCTOBER 15, 12 NOON

Toastmaster: National Director L. A. O'Leary, Head, Chemical Engineering and Research Dept., W. P. Fuller & Co., South San Francisco.

Guest Speaker: Frank E. Clark, Head, Chemical Engineering Division, U. S. Naval Engineering Experiment Station, Annapolis, Md.

Subject: "Old Wine in New Bottles—A New Look at the Substance Water"[5]

Collaborating Committee: Committee D-19 on Industrial Water

Cement and Concrete Industry Luncheon THURSDAY, OCTOBER 15, 12 NOON

Toastmaster: Past President K. B. Woods, Head, School of Civil Engineering and Director, Joint Highway Research Project, Purdue University, Lafayette, Ind.

Guest Speaker: Felix Candela, Architect, Cubiertas Ala S.A., Mexico

Subject: "The Moment of Truth. Adventure in Full Scale Shell Tests"

Collaborating Committees: Committee C-1 on Cement; Committee C-9 on Concrete Aggregates, Committee C-13 on Concrete Pipe

TECHNICAL SESSIONS

SESSION ON PAINT AND RELATED PRODUCTS[6]

MONDAY, OCTOBER 12, 8:30 AM

(*Sponsored by Committee D-1 on Paint, Varnish, Lacquer and Related Products*)

Presiding Officer: National Director L. A. O'Leary, W. P. Fuller & Co.

The Measurement of Viscosity of Inks:

Use of Stormer Viscometer for Testing Rotogravure and Flexographic Inks—Ross Cummings, The California Ink Co., Inc.

Use of a Modified McMichael Viscometer for Consistency of Printing Inks and Ink Vehicles—Ross Cummings, The California Ink Co., Inc.

Water Permeability Studies on Latex and Latex Paint Film—R. C. Simon, The Dow Chemical Co.

Exposure Fence Testing of Metal Protective Paints—C. A. Lominska, National Lead Co.

Lightfastness Testing of Pigment Colors—W. F. Spengeman and G. Wormald, E. I. du Pont de Nemours and Co., Inc.

Discussion of the work of Committee D-1—W. A. Gloger, National Lead Co.

---

[3] Published in the ASTM BULLETIN, No. 242, Dec., 1959, p. 20.
[4] Published in the ASTM BULLETIN, No. 242, Dec., 1959, p. 16.
[5] Published in the ASTM BULLETIN, No. 243, Jan., 1960, p. 17.
[6] Place of publication undetermined.

SYMPOSIUM ON MASONRY MATERIALS[6]

MONDAY, OCTOBER 12, 8:30 AM

(Sponsored by Committees C-12 on Mortars for Unit Masonry and C-15 on Manufactured Masonry Units)

Presiding Officers: Byron P. Weintz (Chairman, Southern California District Council), Consolidated Rock Products Co.
R. W. Harrington, Clay Brick and Tile Assn.
Technical Chairman: M. H. Allen, Structural Clay Products Research Foundation

Materials for Reinforced Masonry Construction—Albyn Mackintosh, Mackintosh and Mackintosh.
Testing Masonry Mortar for Efflorescence—P. L. Rogers, Riverton Lime and Stone Co.
Relation of Current ASTM Specifications to Properties of Lightweight Clay Masonry Units—P. V. Johnson and W. K. Soderstrum, Structural Clay Products Research Foundation.
Field Test Specimens of Mortar and Grout for Reinforced Grouted Brick Masonry—N. W. Kelch, Brick Manufacturers Association of Southern California.
Structural Bond of Mortar and Grout to Concrete Block—R. E. Copeland, National Concrete Masonry Assn.

SYMPOSIUM ON CERAMICS IN NUCLEAR ENERGY[7]

Two Sessions

MONDAY, OCTOBER 12, 8:30 AM

(Sponsored by Committee C-21 on Ceramic Whitewares and Related Products)

Presiding Officer: M. C. Poulsen, Port Costa Brick Works
Technical Chairman: E. D. Lynch, Argonne National Laboratory

Beryllium Oxide for Nuclear Applications—J. E. Hove, Atomics International.
Some Refractory Uranium Compounds—Preparation and Properties—M. J. Snyder, Battelle Memorial Institute.
Dry Pressing Ceramic Fuel Elements to Close Tolerances—F. J. Hartwig, Babcock and Wilcox Co.
Forming of Ceramic Particles for Nuclear Fuels, Control and Burnable Poison Dispersion Components—G. L. Ploetz and A. T. Muccigrosso, General Electric Co.

(Continued at 2:30 pm)

SYMPOSIUM ON NONDESTRUCTIVE TESTING IN THE MISSILE INDUSTRY[8]

Two Sessions

MONDAY, OCTOBER 12, 8:30 AM

(Sponsored by Committee E-7 on Nondestructive Testing)

Presiding Officer: K. R. Jackman, Convair
Technical Chairman: Gerold Tenney, Los Alamos Scientific Laboratory

Radiography of Large Solid Propellant Rocket Motors—E. L. Criscuolo, D. Polansky, J. Halloway and C. H. Dyer, Naval Ordnance Laboratory.
Mobile Field Testing of Missiles and Aircraft—A. Barath and D. J. Hagemaier, Douglas Aircraft Co., Inc.
High-Energy Radiography in the 6-to 30-Mev Range—J. H. Bly and E. A. Burrill, High Voltage Engineering Corp.
Radiography of Weldments in Motion—W. C. Hitt and D. J. Hagemeier, Douglas Aircraft Co., Inc.

(continued at 2:30 pm)

---

[7] To be issued as separate publication *ASTM STP No. 276.*
[8] To be issued as separate publication *ASTM STP No. 278.*

SYMPOSIUM ON SPECTROSCOPY[9]

Six Sessions

MONDAY, OCTOBER 12, 8:30 AM

(Sponsored by Committees E-2 on Emission Spectroscopy and E-13 on Absorption Spectroscopy)

I. Mainly Optical Emission

Presiding Officer: National Director C. L. Clark, The Timken Roller Bearing Co.
Technical Chairman: M. F. Hasler, Applied Research Laboratories

Challenges in Atomic Spectroscopy—J. R. McNally, Jr., Oak Ridge National Laboratory.
Spectroscopy of Radioactive Materials—John Conway, Lawrence Radiation Laboratory, University of California.
Communications in Spectrochemical Analysis—D. L. Fry, General Motors Corp.

(Continued at 2:00 pm Monday; 8:30 am and 2:30 pm Tuesday; and 8:30 am and 2:30 pm Thursday)

SYMPOSIUM ON METHODS FOR TESTING BUILDING CONSTRUCTIONS[10]

MONDAY, OCTOBER 12, 2:30 PM

(Sponsored by Committee E-6 on Methods of Testing Building Constructions)

Presiding Officer: R. F. Legget (Chairman, Committee E-6), National Research Council of Canada
Technical Chairman: N. W. Kelch, Brick Manufacturers Association of Southern California

Introduction—R. F. Legget, National Research Council of Canada.
Testing Models of a Large Wood Roof Beam—J. B. Snodgrass, Forest Products Research Center.
The Testing of Diaphragms to Determine Adequacy in Resisting Horizontal Forces—S. B. Barnes, S. B. Barnes and Associates.
Testing of Large Diaphragms—R. D. Cousineau, Converse Foundation Engineering Co.
Lateral Shear Tests of a Light Gage Steel Building—J. J. Holstein, Structural Engineer.[6]
Determining the Useful Strength of Yard Lumber of the Common Grade—T. K. May, West Coast Lumbermen's Assn.
Testing Reinforced Grouted Brick Masonry and Also Hollow Concrete Masonry Wall Panels to Determine Values in Diagonal Tension and Shear—N. W. Kelch, Brick Manufacturers Association of Southern California.[6]

SYMPOSIUM ON CERAMICS IN NUCLEAR ENERGY[7] *(Continued)*

MONDAY, OCTOBER 12, 2:30 PM

Presiding Officer: C. R. Sutton (Member, Administrative Committee on Papers and Publications), International Nickel Co., Inc.
Technical Chairman: R. M. Fulrath, University of California

Fabrication and Evaluation of Urania-Alumina Fuel Elements and Boron Carbide Burnable Poisons—L. G. Wisnyi, General Electric Co., and K. M. Taylor, The Carborundum Co.
Silicon Carbide Clad-Graphite Matrix Fuel Elements—K. M. Taylor and C. H. McMurtry, The Carborundum Co.
Graphite as a Fuel Matrix—W. C. Riley, Battelle Memorial Institute.
The Fabrication of U_3O_8-Al Dispersion Fuel Elements by Extrusion—R. A. Noland, D. E. Walker and L. C. Hymes, Argonne National Laboratory.

---

[9] To be issued as separate publication *ASTM STP No. 269.*
[10] To be issued as separate publication *ASTM STP No. 275.*

SYMPOSIUM ON NONDESTRUCTIVE TESTING IN THE MISSILE INDUSTRY[8] (*Continued*)

MONDAY, OCTOBER 12, 2:30 PM

Presiding Officer: National Director I. V. Williams, Bell Telephone Laboratories, Inc.
Technical Chairman: Jack Cusick, Naval Ammunition Depot

Ultrasonic Inspection of Adhesion Bonded Aluminum Sandwich Structures—C. C. Kammerer, North American Aviation, Inc.
An Ultrasonic Method to Detect, Count and Measure Particle Size of Contamination in Fluids—C. P. Albertson, Grumman Aircraft Co.
Ultrasonic Gaging of Missile Components During Manufacture—Peter Bloch, Branson Instruments.
Ultrasonic Standards for the Evaluation of Missile Materials and Components—Charles Adams, Rocketdyne, North American Aviation, Inc.

SYMPOSIUM ON ELECTRICAL INSULATING MATERIALS[6]

MONDAY, OCTOBER 12, 2:30 PM

(*Sponsored by Committee D-9 on Electrical Insulating Materials*)

Presiding Officer: Don Bowers, General Petroleum Corp.
Technical Chairman: H. S. Endicott, General Electric Co.

Development of Improved Insulating Oils—N. W. Furby and F. J. Hanly, California Research Corp.
Compatibility of Magnet Wire Insulations and Epoxy Encapsulating Resins—Henry Lee, The Epoxylite Corp.
Activities of the International Electrotechnical Commission Dealing with Electrical Insulating Materials—A. H. Scott, National Bureau of Standards.
Current Activities of Committee D-9—H. S. Endicott, General Electric Co.

SYMPOSIUM ON SPECTROSCOPY[9] (*Continued*)

MONDAY, OCTOBER 12, 2:30 PM

II. X-ray

Presiding Officer: R. E. Paine (Vice-Chairman, Southern California District Council), Aluminum Company of America
Technical Chairman: Allan Zalkin, Lawrence Radiation Laboratory, University of California

Basic Practices in X-Ray Fluorescence—L. S. Birks, U. S. Naval Research Laboratory.
The Future of X-Ray Fluorescence Instrumentation—J. W. Kemp, Applied Research Laboratories, Inc.
Application of X-Ray Spectroscopy to Unsolved Problems in Geochemistry—Isidore Adler, U. S. Geological Survey.
Quantitative Light Element Analysis, Fe to Mg, for Portland Cement by X-Ray Spectrography—E. A. Curley, Riverside Cement Co.

(*Continued at 8:30 am and 2:30 pm Tuesday and 8:30 am and 2:30 pm Thursday*)

SYMPOSIUM ON APPLIED RADIATION AND RADIOISOTOPE TEST METHODS[11]

Two Sessions

TUESDAY, OCTOBER 13, 8:30 AM

(Sponsored by Committee E-10 on Radioisotopes and Radiation Effects)

Presiding Officer: C. F. Fitzwilson, Columbia-Geneva Steel
Technical Chairman: O. M. Bizzell, Atomic Energy Commission

Radioactive Ring Wear Testing in Railroad Diesel Locomotives—C. F. Jursch, Southern Pacific Railroad Co., P. L. Pinotti, Standard Oil Company of California, and D. R. Jones, California Research Corp.

Grain Boundary Segregation Studies by Activation—W. W. Schultz, General Electric Co.

Removal of Radiotagged Fatty Soil From a Glass Surface—J. C. Harris and R. M. Anderson, Monsanto Chemical Co.

Calcium-45-Tagged Clay as a Detergency Test Soil—J. W. Hensley and C. G. Inks, Wyandotte Chemical Corp.

Measurement of Moisture and Density in Soils by the Nuclear Method—John Kuranz, Nuclear-Chicago Corp.

Instrumental Methods of Analysis—G. G. Manov and L. J. Beaufait, Tracerlab, Inc.

(Continued at 2:30 pm)

SYMPOSIUM ON DURABILITY AND WEATHERING OF STRUCTURAL SANDWICH CONSTRUCTIONS[12]

Two Sessions

TUESDAY, OCTOBER 13, 8:30 AM

(Sponsored by Committee C-19 on Structural Sandwich Constructions)

Presiding Officer: National Director R. W. Seniff, Baltimore & Ohio Railroad Co.
Technical Chairman: J. P. Reese, The Martin Co.

Effect of Normal Aging on Physical Properties of Glass Fabric-Plastic Honeycomb Core—A. C. Marshall, Hexcel Products, Inc.

Effect of Natural Environment on Sandwich Structure Fabricated Using AMS 3722 Paper Honeycomb Core—R. F. Zemer, Douglas Aircraft Co.

Effect of Environmental Exposure on Adhesive Bonded Structures—Arnold Tuckerman, Northrup Corp.

Service Experience of Sandwich Construction—Some Approaches to Problems in Aircraft Applications—J. P. Reese, The Martin Co.

(Continued at 2:30 pm)

SYMPOSIUM ON HYDRAULIC FLUIDS[13]

Two Sessions

TUESDAY, OCTOBER 13, 8:30 AM

(Sponsored by Technical Committee N on Hydraulic Fluids of Committee D-2 on Petroleum Products and Lubricants)

I. Problems and Trends in the Use of Hydraulic Fluids

Presiding Officer: National Director H. M. Hancock, The Atlantic Refining Co.
Technical Chairman: L. W. Manley, Socony Mobil Oil Co., Inc.

Objectives and Activities of Technical Committee N—W. H. Millett, Union Carbide Chemicals Co.

---

[11] To be issued as separate publication *ASTM STP No. 268.*

[12] To be issued as separate publication *ASTM STP No. 270.*

[13] To be issued as separate publication *ASTM STP No. 267.*

Are Hydraulics Holding Their Own?—R. Q. Sharpe and K. G. Henrikson, Socony Mobil Oil Co., Inc.
Automotive Central Hydraulic Systems and Their Fluid Requirements—T. H. Risk, Ford Motor Co.
Is Hydraulics Out of This World—G. R. Keller, Autonetics.

(Continued at 2:30 pm)

SYMPOSIUM ON SPECTROSCOPY[9] *(Continued)*

TUESDAY, OCTOBER 13, 8:30 AM

III. Ultraviolet Absorption and Flame Photometry

Technical Chairman: Joseph Guffy, California Research Corp.

Problems in Ultraviolet Absorption Spectroscopy—J. M. Vandenbelt, Parke, Davis & Co.
Spectroscopy in the Region 175–200 mμ—W. I. Kaye, Beckman Instruments Co.
Flame Photometry from Medical and Biological Standpoints—R. E. Thiers, Harvard Medical School.
Analytical Flame Photometry: New Developments—P. T. Gilbert, Jr., Beckman Instruments Co.
Special Problems in the Determination of Tetraethyllead in Gasoline by Flame Photometry—B. E. Buell, Union Oil Co.

(Continued at 2:30 pm Tuesday and 8:30 am and 2:30 pm Thursday)

SYMPOSIUM ON MATERIALS IN THE ELECTRONICS INDUSTRY[14]

TUESDAY, OCTOBER 13, 8:30 AM

Presiding Officer: Roy Henning, Eitel-McCullough, Inc.
Technical Chairman: J. H. Koenig (National Director, ASTM), Rutgers University

Speakers in this session representing four of the ASTM main technical committees will outline the Society's activities toward solution of industry problems in the electronics field. The panel discussion following the papers will provide an opportunity for questions and to discover new areas for ASTM activity.

Magnetic Materials and Testing—W. S. Eberly (Committee A-6 on Magnetic Properties), Carpenter Steel Co.
Electronic Ceramics—J. H. Koenig (Committee C-21 on Ceramic Whitewares and Related Products), Chairman, Electronics Div., American Ceramic Society.
Insulation and Printed Wiring—H. S. Endicott (Committee D-9 on Electrical Insulating Materials), General Electric Co.
Electron-Tube and Semiconductor Materials—S. A. Standing, Raytheon Corp., and F. J. Biondi, Bell Telephone Laboratories, Inc. (Committee F-1 on Materials for Electron Tubes and Semiconductor Devices).

Round Table Discussion

Panel Members: F. J. Biondi, W. S. Eberly, H. S. Endicott, Roy Henning, J. H. Koenig, Frank Y. Speight (ASTM Staff), S. A. Standing.

SYMPOSIUM ON APPLIED RADIATION AND RADIOISOTOPE TEST METHODS[11] *(Continued)*

TUESDAY, OCTOBER 13, 2:30 PM

Technical Chairman: Bruce Ashcraft, Westinghouse Electric Co.

A Radiometric Method for the Determination of Magnesium Oxide in Portland Cement—

---

[14] Not to be published.

J. E. Howes, Jr., C. T. Brown, D. N. Sunderman, M. Pobereskin, and P. Schall, Battelle Memorial Institute.

Tracer Methods for Rapid Determination of the Uniformity of Mixing—R. M. Main, Tracerlab, Inc.

Tracer Techniques for Very Thin Vacuum Evaporated Metal Films—L. E. Preuss, Edsel B. Ford Institute for Medical Research.

Permeability Tests for Organic Sheet Materials—M. Pobereskin, Battelle Memorial Institute.

Radioisotope Methods of Testing the Uniformity of Coated Fabrics—G. B. Foster, Industrial Nucleonics Corp.

SYMPOSIUM ON DURABILITY AND WEATHERING OF STRUCTURAL SANDWICH CONSTRUCTIONS[12] (*Continued*)

TUESDAY, OCTOBER 13, 2:30 PM

Technical Chairman: R. F. Zemer, Douglas Aircraft Co., Inc.

Temperature Rise in Structures Due to Solar Heating—D. K. Rider, Bell Telephone Laboratories, Inc.

Effect of Climatic Variations on the Performance of Polystyrene Foam Core Sandwich Panels in Roofs and Walls—E. A. Edberg, Koppers Co.

Some Experiences and Aspects of Structural Sandwich Applications in Building Construction—R. A. Biggs, Union Carbide Metals Co.

SYMPOSIUM ON HYDRAULIC FLUIDS[13] (*Continued*)

TUESDAY, OCTOBER 13, 2:30 PM

II. Hydraulic Fluid Developments and Evaluation Methods

Presiding Officer: National Director H. D. Wilde, Humble Oil & Refining Co.
Technical Chairman: R. C. Vollmar, Standard Oil Co. of California

Spray Flammability Test for Hydraulic Fluids—H. H. Rowand, Aluminum Company of America.

Hydraulic Fluids for Use in High Pressure Shipboard Equipment—H. F. King and J. A. Coil, Bureau of Ships.

New Developments in High Performance Fluids for Military Aircraft and Commercial Industrial Applications—T. G. Smith, Celanese Corporation of America.

Emulsion Type Fire Resistant Industrial Hydraulic Fluids—C. E. Francis and H. E. Sipple, Shell Oil Co.

Development and Testing of Aircraft Hydraulic Fluids—R. L. Peeler and S. A. Kovacich, California Research Corp.

SYMPOSIUM ON SPECTROSCOPY[9] (*Continued*)

TUESDAY, OCTOBER 13, 2:30 PM

IV. Mainly Magnetic Resonance

Technical Chairman: George Pake, Stanford University

Nuclear Magnetic Resonance Spectroscopy—J. N. Shoolery, Varian Associates.

The Unpaired Electron via EPR—R. H. Sands, University of Michigan.

Electron Spin Resonance Spectra of Free Radicals Derived from Some Polynuclear Heteroaromatic Compounds—L. D. Tuck and D. W. Schieser, University of California.

Maser Applications and Traveling-Wave Techniques for Magnetic Resonance Spectroscopy—A. E. Siegman, Stanford University.

Band Nomenclature for the Ultraviolet Spectra of Conjugated Organic Compounds—F. W. Forbes, Memorial University of Newfoundland, and R. Shilton, England.

(*Continued at 8:30 am and 2:30 pm Thursday*)

SYMPOSIUM ON TREATED WOOD FOR MARINE USE[10]

TUESDAY, OCTOBER 13, 2:30 PM

(*Sponsored by Committee D-7 on Wood*)

Presiding Officer: C. M. Wakeman (Honorary Vice-Chairman, W. C. Committee), Los Angeles Harbor Department

Technical Co-Chairmen: Past-President L. J. Markwardt, U. S. Forest Products Laboratory
L. N. Ericksen, U. S. Forest Service, Forest Utilization Research Division

Use of Small Panels in the Screening of Potential Wood Preservatives—Thorndyke Roe and Harry Hochman, U. S. Naval Civil Engineering Laboratory.

A Standard Procedure for Evaluating the Toxicity of Chemical Agents to the Marine Borer Limnoria Tripunctata—H. P. Vind and Harry Hochman, U. S. Naval Civil Engineering Laboratory.

The Use of Column Chromatography in the Analysis of Creosote—R. W. Drisko and Harry Hochman, U. S. Naval Civil Engineering Laboratory.

A Proposed Standard Rating System for the Evaluation of Wood Piles in Sea Water—E. V. Dockweiler and H. E. Stover, Los Angeles Harbor Department.

Extending the Life of Wood Piles in Sea Water—C. M. Wakeman and L. L. Whiteneck, Los Angeles Harbor Department.

Determining Preservative Retention in Piling by the Assay of Borings—R. H. Baechler, U. S. Forest Products Laboratory.

Marine Exposure Tests of Pressure-Treated Douglas Fir and Southern Pine—R. D. Graham and D. J. Miller, Oregon Forest Products Research Center.

NUCLEAR PROBLEMS FORUM

TUESDAY, OCTOBER 13, 2:30 PM

The Second Forum sponsored by the ASTM Special Administrative Committee on Nuclear Problems publicized the work of the Society to people engaged in nuclear energy projects. Information was exchanged on standardization work being done in the ASTM committees and on work which should be considered. The discussion in the first forum held at the Annual Meeting in June, 1959, was used as a basis for this second forum.

SYMPOSIUM ON EFFECT OF WATER-REDUCING AND SET-RETARDING ADMIXTURES ON PROPERTIES OF CONCRETE[15]

Two Sessions

WEDNESDAY, OCTOBER 14, 8:30 AM

(*Sponsored by Committees C-1 on Cement and C-9 on Concrete and Concrete Aggregates*)

Presiding Officer: Vice-President M. N. Clair, Thompson-Lichtner Co., Inc.

Technical Chairman: R. E. Davis, University of California

Introduction—Bruce Foster, National Bureau of Standards.

Actions of Calcium Sulfate and Admixtures in Portland Cement Pastes—W. C. Hansen, Universal Atlas Cement Co.

Structural and Lean Mass Concrete as Affected by Water-Reducing, Set-Retarding Agents—G. B. Wallace and E. L. Ore, U. S. Bureau of Reclamation.

Observations in Testing and Use of Water-Reducing Retarders—L. H. Tuthill, R. F. Adams, and J. M. Hemme, California State Department of Water Resources.

Effect of Water-Reducing Admixtures and Set-Retarding Admixtures as Influenced by Portland Cement Composition—Milos Polivka and Alexander Klein, University of California.

Field Experience Using Water Reducers in Ready Mixed Concrete—E. L. Howard, Pacific Cement and Aggregates, Inc.

(*Continued at 2:30 pm*)

---

[15] To be issued as separate publication *ASTM STP No. 266.*

SYMPOSIUM ON RADIATION EFFECTS AND DOSIMETRY[7]

Three Sessions

WEDNESDAY, OCTOBER 14, 8:30 AM

(Sponsored by Committee E-10 on Radioisotopes and Radiation Effects)

Presiding Officer: D. W. McLenegan, Hanford Works, General Electric Co.
Technical Chairman: G.W. Pomeroy, General Electric Co., Nuclear Propulsion Dept.

Introduction—V. P. Calkins, General Electric Co.
The Radiochemical Determination of Uranium Burnup—D. N. Sunderman, Battelle Memorial Institute.
Recent Developments in Gamma Dosimetry—S. I. Taimuty, Stanford Research Institute.
Techniques for Measuring Reactor Neutron Spectra—J. B. Trice, General Electric Co.
Reactor Monitoring by Calorimetry—K. L. Hall, R. F. Klaver, J. G. Carroll, and R. O. Bolt, California Research Corp.

(Continued at 2:30 pm Wednesday and 8:30 am Thursday)

SYMPOSIUM ON WOOD IN BUILDING CONSTRUCTIONS[10]

Two Sessions

WEDNESDAY, OCTOBER 14, 8:30 AM

(Sponsored by Committee D-7 on Wood)

Presiding Officer: Past-President L. J. Markwardt (Chairman of Committee D-7), U. S. Forest Products Laboratory
Technical Chairman: Ernst Maag, California State Department of Public Works

Need for Special Engineering Education in the Field of Structural Timber Design—R. A. Glaze, Rilco Laminated Products, Inc.
Quality Control and Inspection—F. J. Hanrahan, American Institute of Timber Construction.
Deflection Limitations as a Factor in Structural Timber Design—R. E. Eby, Rilco Laminated Products, Inc.
Strength Studies of Scarf Joints in Laminated Wood Beams—Jack Longworth, University of Alberta, and C. K. A. Stieda, Wilson Concrete Products Ltd.
Screw-Holding Ability of Western Woods: Effects of Test Variables—J. W. Johnson, Forest Products Research Center.

(Continued at 2:30 pm)

SYMPOSIUM ON FATIGUE OF AIRCRAFT STRUCTURES[16]

Three Sessions

WEDNESDAY, OCTOBER 14, 8:30 AM

(Sponsored by Committee E-9 on Fatigue)

Presiding Officer: National Director R. E. Peterson; (Chairman, Committee E-9), Westinghouse Electric Corp.
Technical Chairman: H. F. Hardrath, National Advisory Committee for Aeronautics

Quantitative and Systematic Attack on Fatigue—E. T. Haire, The Martin Co.
Development of Loading Spectra for Testing Aircraft Components and Structures—Alex McCulloch, Lockheed Aircraft Co.
A Test Method for Sonic Fatigue—R. H. Vreeland, Douglas Aircraft Co.
Low-Cycle Fatigue Strength of Pressurized Components—Joseph Padlog, Space Technological Laboratory, and Ivan Rattinger, Bell Aircraft Corp.

(Continued at 2:30 pm Wednesday and 8:30 am Thursday)

---

[16] To be issued as separate publication *ASTM STP No. 274.*

SYMPOSIUM ON REINFORCED PLASTICS[17]

WEDNESDAY, OCTOBER 14, 8:30 AM

(Sponsored by Committee D-20 on Plastics)

Presiding Officer: P. V. Garin, Southern Pacific Co.
Technical Chairman: National Director A. E. Juve, The B. F. Goodrich Co.

Introductory Remarks—Problems in Testing and Specifying Reinforced Plastics—H. A. Perry, Naval Ordnance Laboratory.

Test Procedure for Obtaining Mechanical Properties of Filament Wound Materials—W. R. McCarthy, Brunswick-Balke-Collender Co.

Problems in Evaluating Reinforcements—I. Silver, P. W. Erickson and H. A. Perry, Naval Ordnance Laboratory.

Permeability of Materials Under High Pressures and at Various Temperatures—G. Epstein and W. W. Gary, Jr., Aerojet-General Corp.

Evaluation of Reinforced Plastics for High Temperature Structural Applications—V. F. Hribar and A. M. Dowell, Hughes Aircraft Co.

SYMPOSIUM ON EFFECT OF WATER-REDUCING AND SET-RETARDING ADMIXTURES ON PROPERTIES OF CONCRETE[15] *(Continued)*

WEDNESDAY, OCTOBER 14, 2:30 PM

Technical Chairman: Milos Polivka, University of California

Detection of Lignosulfonate Retarder in Cement Suspensions and Pastes—T. Thorvaldson and E. G. Swenson, National Research Council of Canada.

Introduction to Producers' Papers on Water-Reducing Admixtures and Set-Retarding Admixtures for Concrete—M. R. Prior and A. B. Adams, Dewey and Almy Chemical Co.

Effect of Water-Reducing Admixtures and Set-Retarding Admixtures on the Properties of Plastic Concrete—C. A. Vollick, Sika Chemical Corp.

The Effect of Water-Reducing Admixtures and Set-Retarding Admixtures on the Properties of Hardened Concrete—D. R. MacPherson and H. C. Fischer, Johns-Manville Products Corp.

Water-Reducing Admixtures and Set-Retarding Admixtures for Concrete: Uses; Specifications; Research Objectives—R. C. Mielenz, Master Builders Co.

Summary—Bruce Foster, National Bureau of Standards.

SYMPOSIUM ON RADIATION EFFECTS AND DOSIMETRY[7] *(Continued)*

WEDNESDAY, OCTOBER 14, 2:30 PM

Technical Chairman: C. R. Sutton, Member, Administrative Committee on Papers and Publications, International Nickel Co., Inc.

Removal Dose as an Environmental Measurement of X-Rays and Gamma Rays—R. L. Hickmott, Wright Patterson Air Force Base.

Dosimetry Nomenclature in the United States—A. M. Tschaeche, Reynolds Electric and Engineering Co.

Dosimetry in Europe and the USSR—J. H. Guill and J. Moteff, General Electric Co.

Radiation Effects Problems in Unmanned Nuclear Propelled Vehicles—G. L. Stiehl, Jr., Convair.

The Effects of Gamma Radiation on Some Electrical Properties of Teflon—W. E. Loy, Jr., The Martin Co.

(Continued at 8:30 am Thursday)

---

[17] To be issued as separate publication *ASTM STP No. 279.*

SYMPOSIUM ON WOOD IN BUILDING CONSTRUCTIONS[10] (*Continued*)

WEDNESDAY, OCTOBER 14, 2:30 PM

Technical Chairman: Ben Benioff, King, Benioff & Associates

The Renaissance of Wood in School Building Construction in California—Ernst Maag, California State Department of Public Works.
Termite Control in California Residential Construction—Walter Ebeling, University of California.
Wood Pole-Type Buildings—W. D. Keeney, American Wood Preservers Institute.
Strength and Related Properties of Wood Poles—E. C. O. Erickson and L. W. Wood, U. S. Forest Products Laboratory.

SYMPOSIUM ON FATIGUE OF AIRCRAFT STRUCTURES[16] (*Continued*)

WEDNESDAY, OCTOBER 14, 2:30 PM

Presiding Officer: H. D. Moran, Aerospace Industries Association of America
Technical Chairman: F. M. Howell, Aluminum Company of America

Development Fatigue Testing of a Helicopter Rotor Blade—A. A. Lischer, McDonnell Aircraft Corp.
Production Methods of Cold Working Joints Subjected to Fretting for Improvement of Fatigue Strength—K. T. Waters, Vertol Aircraft Corp.
Designing Fatigue Resistant Structures—C. R. Smith, Convair.
A Method for Predicting the Rate of Fatigue Crack Propagation—A. J. McEvily, Jr., and Walter Illg, National Aeronautics and Space Administration.

(*Continued at 8:30 am Thursday*)

SYMPOSIUM ON RADIATION EFFECTS AND DOSIMETRY[7] (*Continued*)

THURSDAY, OCTOBER 15, 8:30 AM

Presiding Officer: E. W. Gardiner, California Research Corp.
Technical Chairman: S. Wenk, Applied Radiation Corp.

Standardization of Terminology for Gamma and Electron Beam Radiation Sources—G. E. Danald, U. S. Army Chemical Corp. School.
Atomistic Interpretation of Radiation Effects in Metals—A. Sosin, Atomics International.
Irradiation of Some Pressure Vessel Steels—L. P. Trudeau, International Nickel Co. of Canada, Ltd.
Neutron Radiation Embrittlement at 500 F and 650 F of Reactor Pressure Vessel Steels—J. V. Alger and L. M. Skupien, Westinghouse Electric Co.
Dynamic Radiation Effects Testing Methods—D. M. Newell, E. E. Kerlin, R. R. Bauerlein and R. F. Barrows, Convair.
Concluding Remarks—A. N. Holden, General Electric Co.

SYMPOSIUM ON ROAD AND PAVING MATERIALS[18]

Two Sessions

THURSDAY, OCTOBER 15, 8:30 AM

(*Sponsored by Committee D-4 on Road and Paving Materials*)

I. Symposium on the Zaca-Wigmore Experimental Test Road

Presiding Officer: National Director A. B. Cornthwaite, Virginia Department of Highways
Technical Chairman: B. A. Vallerga, The Asphalt Institute

Progress Report on the Zaca-Wigmore Experimental Asphalt Test Project—F. N. Hveem, Ernst Zube, and J. Skog, California Division of Highways.

---

[18] To be issued as separate publication *ASTM STP No. 277.*

Results of Cooperative Test Series on Asphalts from the Zaca-Wigmore Experimental Project—J. B. Skog, California Division of Highways.

Correlation of Microfilm Durability Tests with Field Hardening Observed on Zaca-Wigmore Experimental Project—W. C. Simpson, T. K. Miles, and R. L. Griffin, Shell Development Co.

(Continued at 2:30 pm)

SYMPOSIUM ON FATIGUE OF AIRCRAFT STRUCTURES[16] *(Continued)*

THURSDAY, OCTOBER 15, 8:30 AM

Presiding Officer: F. T. Wood, Jr., Douglas Aircraft Co.
Technical Chairman: C. R. Smith, Convair

An Observation Concerning the Cycle Ratio in Cumulative Damage—H. J. Grover, Battelle Memorial Institute.

Variable Amplitude Fatigue Tests of Aluminum Alloy Specimens—H. F. Hardrath and E. C. Naumann, National Aeronautics and Space Administration.

Fatigue-Strength Comparisons of Forged Magnesium Alloys—E. H. Schuette, Dow Chemical Co.

Short-Time Compression-Creep Testing of Sheet Utilizing High-Speed Heating—R. W. Fenn, Dow Chemical Co.

SYMPOSIUM ON SPECTROSCOPY[9] *(Continued)*

THURSDAY, OCTOBER 15, 8:30 AM

V. Mainly Infrared Absorption

Technical Chairman: C. S. French, Carnegie Institute of Washington

Some Comments Concerning Quantitative Infrared Spectroscopy—M. K. Wilson and W. C. Steele, Tufts University.

Some of the Problems Facing Chemical Infrared Spectroscopy—W. J. Potts, Dow Chemical Co.

A Technique for Preparing Nonbrittle Solid Organic Samples for I. R. Analyses—L. A. Strait and M. K. Hrenoff, University of California.

Ten Years of Aid to Applied Absorption Spectroscopy by Committee E-13—R. F. Robey, Esso Research and Engineering Co.

(Continued at 2:30 pm)

SYMPOSIUM ON THERMAL ABLATION[17]

Two Sessions

THURSDAY, OCTOBER 15, 8:30 AM

(Sponsored by Committee D-20 on Plastics)

I. Theory and Measurements

Presiding Officer: C. F. Lapier, Matson Navigation Co.
Technical Chairman: H. A. Perry, U. S. Naval Ordnance Laboratory

Introductory Remarks—Ablative Environments—H. A. Perry, Naval Ordnance Laboratory.

Principles and Capabilities of Ablation—W. H. Steurer, Convair.

Arcjet Kinetics Related to Ablation Test Validity—R. D. Buhler, Plasmadyne Corp.

Photographic Technique of Surface Temperature Measurement—J. H. Siviter, Jr., National Aeronautics and Space Administration.

(Continued at 2:30 pm)

SYMPOSIUM ON TECHNICAL DEVELOPMENT IN THE HANDLING AND UTILIZATION OF WATER AND INDUSTRIAL WASTE WATER[19]

Two Sessions

THURSDAY, OCTOBER 15, 8:30 AM

(Sponsored by Committee D-19 on Industrial Water)

Presiding Officer: National Director H. C. Miller, Public Service Electric and Gas Co.
Technical Chairman: W. L. Lamar, U. S. Geological Survey

Disposal of Industrial Radioactive Waste Waters at Hanford—L. C. Schwendiman, R. E. Brown, J. F. Honstead, C. E. Linderoth and D. W. Pearce, General Electric Co.

Determination of Radioactive Materials in Water—F. B. Barker, U. S. Geological Survey.

Utilization of Cooling Towers in Conservation and Pollution Control Programs—J. J. Finnerty, Foster Wheeler Corp.

Improvements in Water Treatment for Once-Through Reactor Cooling—R. B. Richman, General Electric Co.

Disposal of Oil Field Brines in the Central Valley of California—J. M. Morris, Jr., California Department of Water Resources.

(Continued at 2:30 pm)

SYMPOSIUM ON ROAD AND PAVING MATERIALS[18] *(Continued)*

THURSDAY, OCTOBER 15, 2:30 PM

II. General Session

Technical Chairman: F. N. Hveem, California State Department of Public Works.

Effect of Temperature on Flexibility Characteristics of Asphaltic Paving Mixtures—C. L. Monismith, University of California.

Influence of Chemical Composition of Asphalts on Performance, Particularly Durability—F. S. Rostler and R. M. White, Golden Bear Oil Co.

Degradation of Mineral Aggregates—C. E. Minor, Washington State Highway Commission.

A Test for Production of Plastic Fines in the Process of Degradation of Mineral Aggregates—Martin Ekse and H. C. Morris, University of Washington.

SYMPOSIUM ON NEWER METALS[20]

Three Sessions

THURSDAY, OCTOBER 15, 2:30 PM

(Sponsored by Committee B-2 on Non-Ferrous Metals and Alloys

I. Properties of Refractory Metals

Presiding Officer: Jonmore Dickason, Metal Control Laboratories, Inc.
Technical Co-Chairmen: Bruce Gonser, Battelle Memorial Institute
H. P. Croft, Kennecott Copper Corp.

Universal High Temperature Testing Machine for Vacuum or Controlled Atmosphere—M. J. Manjoine, L. L. France and R. T. Begley, Westinghouse Electric Corp.

Mechanical Properties of Molybdenum and Molybdenum-Base Alloy Sheet—M. Semchyshen and R. Q. Barr, Climax Molybdenum Corp.

High Temperature Mechanical Properties of Tantalum—F. C. Holden and R. I. Jaffee, Battelle Memorial Institute, and F. R. Schwartzberg, The Martin Co.

---

[19] To be issued as separate publication *ASTM STP No. 273.*

[20] To be issued as separate publication *ASTM STP No. 272.*

The Effect of Oxygen and Nitrogen on the Workability and Mechanical Properties of Columbium (Niobium)—R. T. Begley and L. L. France, Westinghouse Electric Corp.

Tensile Properties of the Platinum-Group Metals—F. C. Holden, R. W. Douglass and R. I. Jaffee, Battelle Memorial Institute.

(Continued at 8:30 am and 2:30 pm Friday)

SYMPOSIUM ON SPECTROSCOPY[9] *(Continued)*

THURSDAY, OCTOBER 15, 2:30 PM

VI. General Topics in Spectroscopy

Technical Chairman: C. S. French, Carnegie Institute of Washington

The Combination of Methods in the Analysis of Complex Hydrocarbon Systems—N. D. Coggeshall and W. Hubis, Gulf Research and Development Co.

Report on Three European Spectroscopy Meetings Attended Early in 1959—R. R. Brattain, Shell Development Co.

Molecular Spectroscopy in the USSR—D. G. Rea, California Research Corp.

Ultraviolet Spectroscopy: Aromaticity; Absorption Errors—R. A. Friedel, U. S. Bureau of Mines.

SYMPOSIUM ON THERMAL ABLATION[17] *(Continued)*

THURSDAY, OCTOBER 15, 2:30 PM

II. Ablation Tests and Simulation

Presiding Officer: Vice-President A. A. Bates, Portland Cement Assn.
Technical Chairman: George Epstein, Aerojet-General Corp.

Testing of Materials Under Various Ablative Conditions—H. A. King, Aerojet-General Corp.

Part I—Ablation and Erosion in the Rocket Environment and Part II—Ablation and Erosion in the Missile Environment—S. H. Herzog and W. Donaldson, Naval Ordnance Test Station.

Testing of Reinforced Plastics Under Simulated Re-Entry Conditions, Part I—Plasma Jet Calibration—R. R. John and W. M. Bade, AVCO Research and Advanced Development.

Testing of Reinforced Plastics Under Simulated Re-Entry Conditions, Part II—Experimental Determination of the Steady-State Heat of Ablation—R. R. John and H. L. Schick, AVCO Research and Advanced Development.

SYMPOSIUM ON TECHNICAL DEVELOPMENT IN THE HANDLING AND UTILIZATION OF WATER AND INDUSTRIAL WASTE[19] WATER *(Continued)*

THURSDAY, OCTOBER 15, 2:30 PM

Presiding Officer: President F. L. LaQue, International Nickel Co., Inc.
Technical Chairman: L. Drew Betz, Betz Laboratories, Inc.

Cities Can and Must Provide Municipal Waste Treatment, Not Sewage Treatment—J. E. Kinney, Sanitary Engineering Consultant.

Field Testing of Corrosion Inhibitors in Sea Water—C. C. Wright, C. C. Wright Oilwell Research, Inc.

Effect of Industrial Wastes on Waters for Irrigation Use—L. V. Wilcox, U. S. Salinity Laboratory.

Operation of Batch-Type and Continuous Electric Membrane Demineralizers on the Muincipal Water Supply of Coalinga, California—W. E. Katz, Ionics, Inc.

Present Status of Solar Distillation of Saline Water with Particular Regard to Material Problems—R. P. Lappala, L. L. Yaeger, and Johan Bjorksten, Bjorksten Research Laboratories, Inc.

SYMPOSIUM ON ADHESION AND ADHESIVES[21]

THURSDAY, OCTOBER 15, 2:30 PM

(*Sponsored by Subcommittee XI on Metal Bonding Adhesives of Committee D-14 on Adhesives*)

Presiding Officer: G. J. Grieve, Pacific Paint and Varnish Co.
Technical Chairman: Steven Yurenka, Narmco Industries, Inc.

Surface Chemistry of Adhesion—Samuel Muchnick, Consultant.
Lap Shear and Creep Testing of Metal-Adhesive Bonds in Germany—K. F. Hahn, Douglass Aircraft Co.
Elevated Temperature Resistant Metal-to-Metal Adhesives Derived from Organic-Inorganic Polymer Systems—E. C. Janis, Narmco Industries, Inc.
Preliminary Evaluation of Ceramic Adhesives for Stainless Steel—L. E. Gates and W. E. Lent, Hughes Aircraft Co.

SYMPOSIUM ON BITUMINOUS WATERPROOFING AND ROOFING MATERIALS[22]

Two Sessions

FRIDAY, OCTOBER 16, 8:30 AM

(*Sponsored by Committee D-8 on Bituminous Materials for Roofing, Waterproofing and Related Building or Industrial Uses*)

Presiding Officer: F. D. Tuemmler, Shell Development Co.
Technical Chairman: P. E. McCoy, American Bitumuls and Asphalt Co.

This is Your Roofing Asphalt—Composition and Rheology—A. B. Brown and S. W. Sparks, Standard Oil Company of Indiana.
Some Qualitative Effects of Composition and Processing on the Weatherability of Coating Grade Asphalts—E. W. Mertens, California Research Corp., and S. H. Greenfeld, National Bureau of Standards.
Manufacture and Application of Asphalt Roofing—L. C. Haack, Certain-teed Products Corp.

(*Continued at 2:30 pm*)

SYMPOSIUM ON SOILS FOR ENGINEERING PURPOSES[23]

Two Sessions

FRIDAY, OCTOBER 16, 8:30 AM

(*Sponsored by Committee D-18 on Soils for Engineering Purposes*)

Technical Chairman: F. J. Converse, Converse Foundation Engineering

Measurement of Excess Hydrostatic Pressures in Soils—W. G. Weber, Jr., California Division of Highways.
Moisture and Strength Variation in a Thick, Uniform Clay Layer—W. G. Weber, Jr., and W. F. Kleiman, California Division of Highways.
Extended Repetitive Plate Load Tests for the Evaluation and Design of Concrete Pavements—B. B. Gordon, Porter, Urquhart, McCreary and O'Brien.
Some Laboratory Studies of the Moisture-Density Relations of Soils—R. F. Dawson, University of Texas.

(*Continued at 2:30 pm*)

---

[21] To be issued as separate publication *ASTM STP No. 271*.
[22] To be issued as separate publication *ASTM STP No. 280*.
[23] These papers are included in *ASTM STP No. 254*.

SYMPOSIUM ON NEWER METALS (*Continued*)[20]

FRIDAY, OCTOBER 16, 8:30 AM

II. Nuclear and Light Metals

Presiding Officer: National Director W. L. Fink, Aluminum Company of America
Technical Co-Chairmen: R. I. Jaffee, Battelle Memorial Institute
W. W. Stephen, The Carborundum Co.

Effect of Purity and Manufacturing Variables on the Elevated Temperature Properties of Beryllium—W. W. Beaver, R. G. O'Rourke, and J. N. Hurd, Brush Beryllium Co.

The Stress-Rupture and Creep Properties of QMV Beryllium Metal—J. N. Hurd, R. G. O'Rourke, and W. W. Beaver, Brush Beryllium Co.

Test Methods Used for Zirconium and Zirconium Alloy in the Naval Reactor Program—H. L. Kall, Carborundum Metals Co.

The Effects of Heat Treatments on the Tensile and Corrosion Properties of Zircaloy-2—J. G. Goodwin, L. S. Rubenstein, and F. L. Shubert, Westinghouse Electric Corp.

Survey of the Mechanical Properties of Yttrium and Yttrium Alloys—O. N. Carlson, D. W. Bare, E. D. Gibson, and F. A. Schmidt, Iowa State University.

(*Continued at 2:30 pm*)

SYMPOSIUM ON AIR POLLUTION CONTROL[6]

Two Sessions

FRIDAY, OCTOBER 16, 8:30 AM

Presiding Officer: R. J. Wirshing (Member, Administrative Committee on Papers and Publications), General Motors Corp.
Technical Chairman: Moyer Thomas, Stanford Research Institute

The Air Pollution Potential of California Coastal Climate—Elmer Robinson, Bay Area Air Pollution Control District.

Wind and Weather Summaries for Chemical Plant Design and Air Pollution Control—R. J. Moore, T. D. Boscia, and R. W. Lundeen, Dow Chemical Co.

Fluorescent Dyes as Airborne Tracer Materials—H. C. Wohlers, Stanford Research Institute—T. E. Kass and K. R. Johnson, Food Machinery and Chemical Corp.

Determination of Gaseous and Particulate Inorganic Fluorides in the Atmosphere—M. R. Pack, A. C. Hill, and L. G. Transtrum, Columbia Geneva Steel Co.

(*Continued at 2:30 pm*)

SYMPOSIUM ON POSTIRRADIATION EFFECTS IN POLYMERS[7]

FRIDAY, OCTOBER 16, 8:30 AM

(*Sponsored by Subcommittee II on Effects of Nuclear and High Energy Radiation, Committees D-9 on Electrical Insulating Materials and D-20 on Plastics*)

Presiding Officer: T. K. Cleveland, Philadelphia Quartz Company of California
Technical Chairman: D. J. Metz, Brookhaven National Laboratory

The Significance to ASTM of Postirradiation Effects in Irradiated Materials—D. S. Ballantine, Brookhaven National Laboratory.

Electron Spin Resonance Studies of Free Radicals in Irradiated Materials—L. A. Wall, National Bureau of Standards.

Postirradiation Oxidation and Molecular Weight Changes in Polystyrene and Poly-(Methyl Methacrylate)—W. W. Parkinson and D. Binder, Oak Ridge National Laboratory.

Postirradiation Dielectric Properties of Silicones—C. G. Currin, Dow Corning Corp.

Postirradiation Effects: Monomers and Polymers—E. F. Degering, G. J. Caldarella, and M. A. Mancini, U. S. Army Quartermaster Research and Engineering Center.

SYMPOSIUM ON BITUMINOUS WATERPROOFING AND ROOFING MATERIALS[22] *(Continued)*

FRIDAY, OCTOBER 16, 2:30 PM

Technical Chairman: G. E. Manning, Underwriters Laboratory

Coal Tar Flat Roof Pitch—W. F. Fair, Jr., and M. D. Chamberlain, Koppers Co., Inc.
Industrial Pitches—C. U. Pittman, Koppers Co., Inc., and J. W. Donegan, Allied Chemical Corp.
Asphaltic Grouts, Mastics and Cold Applied Cements—W. L. Butterfield and F. H. Haney, Flintkote Co.
Glass Fiber Felt Roofing Products—J. G. Hayes, Owens-Corning Fiberglas Corp.

SYMPOSIUM ON SOILS FOR ENGINEERING PURPOSES[23] *(Continued)*

FRIDAY, OCTOBER 16, 2:30 PM

Presiding Officer: H. de Bussieres, Curtis and Tompkins, Ltd.
Technical Chairman: R. Lundgren, Woodward, Clyde, Sherard and Associates

On Determination of Dynamic Characteristics of Soils *in situ*—R. H. Bernhard, Rutgers State University. (Presented by title only.)
The Effect of Temperature on Moisture Contents as Determined by Centrifuge and Tension Techniques—R. C. Prill and A. I. Johnson, U. S. Geological Survey.
The Effect of Particle Shape and Texture on the Strength of Noncohesive Aggregates—H. C. Morris, University of Washington.
Triaxial Compression Tests on Soils Using Variable Lateral Pressure—R. H. Meese and R. W. Long, University of Washington.
Soils Classification Triangle Based on the Unified Soil Classification System—Jack McMinn, Brewer and Associates.

SYMPOSIUM ON NEWER METALS[20] *(Continued)*

FRIDAY, OCTOBER 16, 2:30 PM

III. Processing of Newer Metals

Presiding Officer: National Director P. A. Archibald, Standard Steel Works
Technical Co-Chairmen: Thomas Tietz, Lockheed Aircraft Corp.
H. W. Highriter, Fansteel Metallurgical Corp.

Columbium and Tantalum Fabrication—H. M. McCullough, Universal-Cyclops Steel Corp.
Deoxidation and Denitrification Studies of Chromium and the Effect on Low Temperature Properties—H. L. Levingston and N. J. Grant, Massachusetts Institute of Technology.
Processing and Properties of Tungsten—C. H. Toensing, General Electric Co.
The Solubility of Nitrogen and Oxygen in Lithium and Methods of Lithium Purification—E. E. Hoffman, Oak Ridge National Laboratory.
Coulometric Determination of Tin with Electrolytically Generated Iodine: Application to Analysis of Zircaloy—H. Freund and R. D. Caton, Oregon State College. (Presented by title only.)

SYMPOSIUM ON AIR POLLUTION CONTROL[6] (*Continued*)

FRIDAY, OCTOBER 16, 2:30 PM

Technical Chairman: J. D. Coons, Bay Area Air Pollution Control District

The Colorimetric Determination of Formaldehyde and Methanol from Combustion Sources—M. Feldstein, Bay Area Air Pollution Control District.

The Ram Strati-Charge Engine—R. M. Heintz and K. Kadoch.

Panel Discussion—Air Pollution Implications of the Ram Strati-Charge Engine—R. M. Heintz, K. Kadoch, and Dale Hutchison, Stanford Research Institute; W. R. Linville, Los Angeles County Air Pollution Control District; and Benjamin Linsky, Bay Area Air Pollution Control District.

SYMPOSIUM ON STANDARDS—ARE CHANGES IN ORDER?[6]

FRIDAY, OCTOBER 16, 2:30 PM

Presiding Officer: George Harnden, General Electric Co.
Technical Co-Chairmen: E. V. Noe, Pacific Gas and Electric Co.
H. A. Williams, Stanford University

Sound Standards Stimulate Manufacturing—Paul McCoy, American Bitumuls and Asphalt Co.

Standardization's Role in the Purchasing Function—W. C. Brunberg, Western Pacific Railroad Co.

Comments on ASTM Standards for Highway Materials—F. N. Hveem, California Department of Public Works.

Some Proposed Changes in Standards Based Upon Research in Materials—Joseph Marin, Pennsylvania State University.

THE IMPACT OF SCIENCE ON ASTM

ANNUAL ADDRESS BY THE PRESIDENT

K. B. WOODS[1]

JUNE 23, 1959

It is a bit trite to state that "new developments in science are of great importance to the work of ASTM." However, this statement becomes quite realistic by making a simple comparison of the status of engineering and scientific developments at the time of the beginning of ASTM with the present status almost 60 years later. In 1900, the telephone was a feeble device; the use of electricity had hardly begun; automobiles were 2-cylinder buggies; and there were no road systems. We still had malaria, typhoid fever, and a multitude of other diseases rarely encountered today.

In contrast, in communications we now have superb telephone systems, radio, and both black-and-white and color television. We have automobiles—almost two cars to the family—and airplanes and automobiles are fast replacing trains as a common method of transporting people. Radar is used for many purposes—the latest to measure with high precision the distance to the planet Venus; we have rockets and guided missiles, and there seems to be little doubt about man conquering at least a portion of outer space in the immediate future.

During this same period, parallel development took place in engineering education. In 1900 we had no schools of aeronautical, industrial, chemical, or metallurgical engineering, and the curricula of civil, mechanical, and electrical engineering were packed with laboratory courses and subject matter now considered in the "skills" category. In 60 years, this concept has been replaced by a scientific-professional one with more and more emphasis on the fundamentals.

The history of ASTM also follows this pattern. At the turn of the century, the research data, standards, specifications, and methods of tests were quite simple—at least when compared with the 14,000-page book of standards released this year.

It is my purpose today to expand on this theme: to examine the expanding role and heightening status of science, engineering curricula, and the ASTM during the last half century. Finally, I shall venture to project the work of ASTM a modest distance into the future and will offer some suggestions for future administrative procedures.

ENGINEERING EDUCATION

An inspection of a 50- or 60-year-old bulletin of any good school of engineering will show, in comparison with 1959 offerings, amazing changes. In one such instance, a 1910 bulletin contains a listing of over 40 semester hours of required material in shop, railroad curves, surveying, and related material required for the B.S.C.E. degree. The 1959 catalog for this same institution contains no requirement for shop, three hours of good-quality mathematical engineering graphics, and six hours of high-quality surveying (in part engineering measurements), much of which is of a theoretical nature. It is probable that these many hours of shop, drawing, and surveying were not especially out of line with the professional needs of the young graduate civil engineer 50 years or more ago.

---

[1] Head, School of Civil Engineering; and Director, Joint Highway Research Project, Purdue University, Lafayette, Ind.

Referring again to the amount of time spent on shop, surveying, and railroad curves through the years, a check on the semester hours of an important civil engineering school shows the following: 1910, 42 hours; 1920, 26 hours; 1930, 21 hours; 1940, 17 hours; 1950, 17 hours; and in 1959, 9 hours. Note the 20-year lag from 1930 to 1950. The great depression in the 1930's appears to have had an influence on the development of engineering curricula because of an apparent slowing down of the process of adding mathematics, physics, and chemistry as replacement material for some of the less important laboratory and field courses.

World War II also played a part in this evolution of engineering curricula, although of a somewhat different character. All segments of the military establishment set up programs in the colleges and universities of the country to provide for general curricula in the engineering professions, as well as specialized programs designed to accommodate students with particular abilities—in electronics, for instance. With World War II also came great advances in the physical sciences, and these in turn provided better tools for many of the great developments during this period and, later, in aeronautical, electrical, chemical, and to some extent in mechanical and metallurgical engineering. The student bodies in engineering during World War II came largely from the military establishment, and there was a rapid turnover. Immediately following the war, the engineering schools were literally swamped with unprecedented numbers of students returning from military service. Thus, it can be seen, largely because of preoccupation of our engineering faculties, both during World War II and for a few years following, that changes in engineering curricula were not as rapid as were the new developments in science. This has been true especially in civil engineering.

In the 1950's, most of us concerned with engineering education have seen tremendous changes in engineering-curricula concepts. Not only have we been trying to catch up with some of the changes we should have made in the 1930's, 1940's, and early 1950's, but at the same time we have been making great efforts to keep pace with current developments. In my opinion engineering in all of its branches has undergone more fundamental changes during the past seven or eight years than had occurred during the previous 30 years or so.

The changes are of two general kinds: the adoption of science-oriented engineering programs, and the development of strong professional or design progams. In both cases, modern concepts of science and of engineering are gradually replacing those courses (and especially laboratory courses) that were originally designed to develop proficiency in skills.

During the current period of great change, it is difficult to project the course of engineering education very far into the future. Certain trends are noteworthy. For instance, the chemical engineer will unquestionably maintain his identity in the years ahead even though substantial changes are taking place in many of his undergraduate and graduate programs.

Again, the curricula in aeronautical and electrical engineering, engineering science, and, in many instances, metallurgical engineering, are generally significantly science-oriented. I stated earlier this week[2] that the future individual engineering identity of the undergraduate programs in these areas is in doubt. In mechanical engineering, similar trends are apparent but not in the same direction or in comparable amounts as in the highly science-oriented programs. In civil engineering, the impact of science has been as significant as in other engineering curricula; however, the civil engineering educators (and perhaps the profession itself) have not responded as quickly as in the other areas. Here, too, however, can be observed very significant changes in many institutions in which both science-oriented as well as high-level professional programs are replacing rather rapidly the programs based on old concepts. I have said on another occasion that "Civil engineering is on the threshold of serving mankind in a manner undreamed of a few years ago. The future is bright, but specialization calls for intensive graduate instruction if we are to have continued progress."[3] I think now that this statement holds for all of engineering. In this same line of reasoning, I said earlier this week "that one of the great deficiencies in American engineering education lies in our inability to attract and stimulate interest in a higher percentage of our good engineering students to pursue work toward an advanced engineering or science degree."

IMPACT OF SCIENCE ON ASTM

In the work of ASTM it is appropriate to make comparisons of progress at different intervals and to attempt a prediction of future activity by a projection of the past and present trends. A study of membership growth and the number of ASTM standards published should be revealing.

Membership Trends:

A check of the 50-year membership data of ASTM shows a rather steady climb from 800 members in 1909 to 4000 in 1930. The influence of the depression on membership is as significant as is the slow progress in modernizing engineering curricula. The membership total of 4000 in 1930 barely reached 4200 ten years later. The influence of scientific developments during World War II is again apparent, with the membership climbing to about 5000 by 1945. From this period to the present, the membership has again increased sharply until we now have about 10,000 members. I think this latest surge of interest is directly related to the sharp increase in the number of major contributions from science from which has resulted not only an improvement in our understanding of basic phenomena but also the development of methods for evaluating the quality of materials. No less important is the great upsurge in the design and production of completely new engineering materials. If one is to assume that there will be a steady and perhaps even an accelerated development in pure sciences, then it appears obvious that there will also be a similar increase in our knowledge of materials and in our ability to design and produce large numbers of entirely new materials.

It follows logically enough, then, that increases in membership in the Society will continue.

Number of ASTM Standards Published:

Again using the 50-year period as a base, it is interesting to note how closely the curve for the number of standards published per year follows the membership curve. For instance, there was a steady increase in the number of standards produced from about 200 in 1919 to a little over 500 in 1929. The influence of the depression also appears here, since the number published by 1939 had reached only something less than 800. The developments in scientific endeavors during and following World War II show during this 20-year period a 300 per cent increase from 800 to almost 2500 standards published. In other words, we have published three times as many standards in the last 20 years as we did during the first 40 years of the life of ASTM.

---

[2] Introduction to Joint ASEE-ASTM Symposium on "Impact of Developments in Materials Sciences on Engineering Education."

[3] *Engineering News-Record*, July 3, 1958.

Here again, one must conclude that the value of the work of the Society will increase in the immediate years ahead and that this rate of increase will be sharply upward. However, in projecting the work of a society such as ASTM, it must be remembered that the history of all professional societies is relatively brief—in fact, it is limited largely to the life of modern technology. Consequently, our experience background is small and prognostications are difficult.

Development of Materials Sciences:

Of equal importance in estimating the future trends of the Society is a study of improvements in our ability to evaluate existing materials; for instance, it would be appropriate this year to base some of our estimates on a study of highway materials since we are in the midst of the nation's great hundred-billion dollar highway program.

In considering all of these broad aspects of highway engineering and indeed of civil engineering itself, none is of greater importance than materials. Included in the highway materials field are bituminous materials, bituminous concrete, cement, portland-cement concrete, aggregates, steel, paint, plastics, chemicals, and soils.

To use soil mechanics as an illustration of the rapid change in our ability to use materials economically in the highway area, it can be seen that within a period of 25 years we have been able to develop methods of evaluation and design, and to control construction from zero level to one of serious scientific importance. As a matter of fact, the use (or nonuse) of the developments that have taken place in the soil materials and related fields in the past 25 years will probably determine to a considerable degree whether success or considerable failures shall be the lot of the currently constructed interstate highway system.

This is only one of many examples of scientific developments on highway materials in other engineering developments. In the field of cement and concrete technology Grinter[4] states, "The art of concrete construction has changed with scientific understanding of the setting process, with change in winter protection procedures, with air entrainment, with water removal techniques, with vibrations, with reusable forms, with precast elements, with lift slabs, with prestress, with fast-moving rubber tired vehicles, etc." To this list could well be added the great advances that have been made in our ability to understand and measure the physical and chemical properties of mineral aggregates, to appraise the factors which influence the durability of concrete, and of the contributions made in the past 15 years on the subject of alkali-aggregate reaction.

By the same token, our knowledge of physical and chemical properties of bituminous materials has been greatly expanded, and we have a better understanding of mix design and of the interrelationships between bitumens and aggregates.

Here again, it is appropriate to observe the impact of science—especially the developments in chemistry and physics—upon highway materials and indeed upon all of the ASTM spectrum of engineering materials. It follows, therefore, that with increased emphasis on the basic sciences in the engineering and science curricula this already heavy impact will increase in the immediate years ahead.

---

[4] L. E. Grinter, "Importance of Emphasis in Civil Engineering Education," *Journal of Professional Practice, Proceedings*, Am. Soc. Civil Engineers, Vol. 84, Dec., 1958.

DESIGN OF NEW MATERIALS

A vivid example of the many future developments of materials and knowledge to be sought concerning present materials can be seen in the statement of problems involved with the flight of a relatively slow-speed B-58 bomber. I have been told[5] that even in this bomber, which has a top Mach number of approximately 3, the material properties requirements due to the temperatures imposed are extremely severe. For example, in a typical B-58 mission in which the aircraft takes off at sea level, obtains Mach number 3 velocity at 30,000 ft elevation, and drops down rapidly in Mach number at a lower altitude, certain portions of the aircraft's structure may reach temperatures as high as 1000 F. One would need to extrapolate beyond this range to determine the more severe conditions presented in missile flights. Such conditions give rise to a variety of structural and material problems. Included among these are: (1) thermal stresses due to nonuniform temperature distribution, such stresses being both static and dynamic in character; (2) changes in material properties due to the high-temperature range; and (3) creep and thermal fatigue.

It is thus seen that while we have made much progress in material technology the future holds even more challenges for us. In general, success in solving the problems of materials will be the key to success in the broader problems introduced by the space age.

DISCUSSION AND CONCLUSIONS

In this presentation, it has been observed that the impact of scientific knowledge has already produced important changes in the materials work of ASTM. The officers and the Board of Directors of the Society have recognized the importance and potentialities of this change to its members. The Board has provided the machinery for a new Materials Science Division which is to provide a forum and a medium for publication of basic knowledge in the field of materials. This move is, perhaps, one of the most important additions to the scope of the Society since its inception.

Summing up the information available covering the probable trends for the Society of the future, it would seem appropriate to emphasize that:

1. The *impact* of scientific developments has been of major importance and this has been especially apparent in the work of the Society in the past 15 years.
2. The *impact* of new developments is likely to be of much greater significance in the future than in the past.
3. The *response* of engineering education to the trend in accelerated developments through science will be of significance to the work of ASTM, especially in the design of new materials.

On the basis of these considerations it is my opinion that the officers, the Board of Directors, and members should continue their efforts to prepare the Society to adjust to greater demands resulting from new developments. The Society must accommodate an ever-increasing membership, provide for more standards for both old and new materials, and arrange for an increasing demand for publication space from all segments of the Society's vast structure, including material from the new Division of Materials Sciences.

---

[5] H. M. DeGroff, "Research in Aeronautical Engineering at Purdue University," address, May 1959.

1959 ANNUAL REPORT OF THE BOARD OF DIRECTORS

PAGE
New Division on Materials Sciences 61
The Society's Name 62
Long-Range Planning 63
Technical Activities 66
National and Other Meetings 69
District and Related Activities 71
Membership 73
Honors and Awards 75
Finances 76
Cooperative Activities 79

PAGE
Appendix I—Report of Auditors 81

Reports of Administrative Committees:

Appendix II—On Standards 95
Appendix III—On Papers and Publications. 102
Appendix IV—On District Activities 106
Appendix V—On Research 107
Appendix VI—On Simulated Service Testing 109

THE PAST year has continued to be one of bustling activity in the Society. Not only have there been a number of important new technical activities instituted and others authorized, but in addition committees working under the Long-Range Planning Committee have begun an exhaustive analysis of our current important operations, including our standardization procedures and our technical committee operations. Further reference is made to LRP studies later in this report.

New Division on Materials Sciences:

Many consider of utmost importance the Board's decision to organize promptly a new Division on Materials Sciences, whose objectives will be to focus attention and coordinate studies and discussions on the fundamentals of materials as distinct from engineering considerations. The scope of the division is to cover all facets of the science of materials including education and research, as well as the collection, establishment, and publication of appropriate information and material, on a periodical basis if found desirable.

This may be the first step in a rearrangement of the organization of the Society into divisions, but this whole subject will be considered further by both the Long-Range Planning Committee and the Board. Some distinct advantages would accrue if we had certain families of technical committees organized as divisions; but because of the number of operational problems the Directors definitely do not wish to create additional administrative or operating groups unless they will simplify our already complicated standards and research activities.

It was felt, however, that a separate division would be desirable to emphasize the Society's long concern with fundamentals. For one thing it will provide a special place for concentrating and bringing together scientists and specialists in different fields. ASTM is in a unique position to provide a forum for materials scientists and engineers, since its interests and long-time activities cut horizontally across the entire materials field. Here there is common ground for the physicist, the chemist, and the engineer in a variety of fields—electrical, mechanical, civil, chemical, and many more.

The team approach representing these many disciplines is not new in ASTM—that is the way the Society's over 80 technical committees have been functioning for many years. Research activities relating to fundamentals under way in many of the committees will continue as now organized. The new division can assist or aid in coordination.

Many feel that if the Society's important work—concentrated on "Research and Standards for Materials"—is to expand and keep pace with scientific developments and provide industry with the essential data and standards needed to continue its healthy growth, there must be organized efforts on fundamentals.

All technical committees have been alerted to the formation and purposes of the new division and their officers are to be kept fully informed of developments. It is not expected there will be any changes in the work of the technical committees, certainly not for some time. The Society's President, Vice-Presidents, and Executive Secretary were authorized to implement the division, and they are extending invitations to a group of men who should be especially concerned to serve on an advisory committee and to meet during the coming Annual Meeting. Further announcements will be made.

The Society's Name:

ASTM is really a society for *materials*. This is recognized by all those who have

HIGHLIGHTS, 1958 FINANCIAL OPERATIONS

	1958	1957	1956
Gross Receipts:			
Membership Dues	$297 800	$283 400	$269 000
Sales of Publications	657 000	623 900	695 950
Other	129 200	113 400	117 600
Total	$1 084 000	$1 020 700	$1 082 550
Expenditures:			
Salaries	$406 100	$360 200	$309 400
Printing	577 030	430 200	342 300
Other	269 300	237 800	244 800
Total	$1 252 400	$1 028 200	$896 500
Favorable Operating Balance	....	....	$186 050
Operating Deficit	$168 400	$7 500	....
Net Assets:			
Real Estate	$281 700	$271 500	$269 450
Investments and Cash	1 238 500	1 251 300	$1 048 315
Equipment	38 800	36 900	32 900
Total	$1 559 000	$1 559 700	$1 350 665
Asset Equity of Each Member	$162	$170	$160
Number of Members	9 612	9 159	8 408
	1958	1957	1956
Pages of Publications	21 443	9 652	9 349
Number of Employees	82	78	75

close working knowledge of the many activities involving both research as well as standardization for materials. The word "testing" in the name American Society for Testing Materials has long been felt to be a problem since there are many phases of the work in the committees which are much broader than "testing." This name was inherited from the Society's forerunner, the International Association for Testing Materials. In 1902, when the original charter was granted for a separate body because of the Americans' desire to work on specification, that name was retained because it was considered in keeping with the concept of purpose and scope of ASTM at that time. Since then the activities have widened immeasurably, and the new Division on Materials Sciences directs attention to an area where much future expansion is anticipated, even though work on fundamentals has been going on in committees for years.

The subject of the Society's name has been discussed informally in the Long-Range Planning Committee and in the Board of Directors, and a special committee appointed by LRP recently conducted a survey of former officers to get their ideas. Two names in particular have been considered as conveying a much better concept of the scope of ASTM: (1) "Association for Standardization and Technology for Materials"; and (2) "American Society for Science and Technology of Materials." Some feel the word "standardization" would be very desirable, giving a much broader connotation than the word "testing"; and since basically the work of ASTM is concerned with advancing a more efficient use of materials the term "technology" is not considered too broad.

On one point there is virtually unanimous opinion, namely, that the initials "ASTM" which mean so much after all these years should be continued no matter what the eventual title might be. This practice has been used by other scientific organizations which have changed their name due to expansion or new concepts of their work, and also by industrial organizations.

No action is called for at the 1959 Annual Meeting but some recommendations might be forthcoming in 1960, since a change in name would involve a modification of the Charter as well as the By-laws.

Long-Range Planning

IT is desirable for any voluntary and service organization as well as an industrial concern occasionally to take stock, to determine what progress has been made, to ascertain what the demands may be for the future, and to evaluate the organizational set-up based on what is expected. This is the purpose for establishing the Long-Range Planning Committee (LRP).

It was evident to LRP from the very start that a number of special committees would need to be established to include men with wide experience in the respective areas needing study if the work were not to be too long drawn out. Consequently the LRP has in the past 18 months, with the approval of the Board of Directors, set up several such groups to evaluate the some 60 specific questions on which answers are desired.

The Committee on Education has submitted its report, and three specific suggestions have been implemented, namely, an Administrative Committee on Education in Materials, a Committee on Fellowships and Grants-in-Aid, and a Division on Materials Sciences.

Two subjects extremely involved but vital to our whole operation are standardization procedures and technical committee operations. Two special committees in these respective fields have been organized and have held meetings. Several additional meetings will be necessary in order to resolve the many questions being studied.

A special committee on individual memberships has been organized to evaluate among other matters a suggestion for a special class of membership open to graduates of technical institutes, junior colleges, and those working in chemical, physical, and related laboratories. Many of these individuals are concerned with the properties of materials from the standpoint of both research and standardization. Another question is whether justification exists for instituting a professional class of membership with specific qualifications, perhaps termed "materials engineer," since there is an apparent trend to grant degrees to either undergraduates or graduates in "materials engineering."

Reference has been made to discussions concerning the name of the Society. Other areas under study include the very broad one of "research;" the Administrative Committee on Research has held discussions and the Board has devoted an evening to consideration of portions of this area. The ACR is holding a Forum on Research at the Annual Meeting to bring out ideas and to clarify, especially in the work of technical committees, what we mean by "research."

Two other very important problems needing coordinated study are (1) publications, including the tremendous growth of the Book of ASTM Standards, with its resulting problems, and (2) the matter of divisionalization. For example, would a division on ferrous metals or non-ferrous metals, or both, with separate meetings and its own standardization coordinating committee, simplify our operations? Or would divisions add further to the burden of the consumer who in many instances must cover a wide range of committee activities?

Revised Charter:

To implement an LRP recommendation that the number of Directors be increased from 15 to 18, the Charter was revised by action at the Annual Meeting and also at a subsequent special meeting of the membership held at ASTM Headquarters on December 17, 1958. By this action, the number of Directors will be governed in the future by the By-laws, thus avoiding the necessity of appealing to the Courts. Brief minutes of the special meeting appear in the 1958 *Proceedings*.

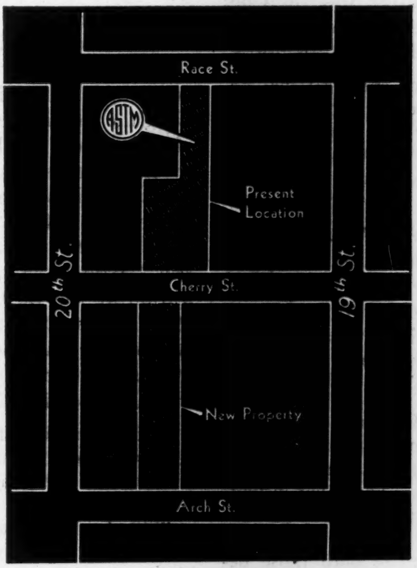
Enlarged Headquarters Building:

A part of the LRP program is an evaluation of headquarters building needs for the next ten years. Since space in the present two buildings on Race Street and Cherry Street would be fully occupied within the next year or two and because the decision on location of a new building could not be reached until a number of other studies are completed, there was considerable sentiment for demolishing the Cherry St. property and erecting thereon a six to eight story building which would serve us for the next 20 years or so. On the other hand proponents of a modern new building and location, perhaps adjacent to one of Philadelphia's expressways, presented strong points to support their view. In the meantime, a property running from Cherry Street to Arch Street, directly opposite our Cherry Street property, became available, and it was found that the building, though old, could be utilized for several years for committee meetings as well as Staff operations. The property was bought, and data on costs are given later in discussions on finances. It was felt that in addition to the sizable ground area and buildings we were also buying some "time." The general location of the new property in relation to the Race Street and Cherry Street properties is shown in the accompanying map. It will be seen from the following table that the floor area of the new Arch Street property is about the same as for the building originally purchased on Race Street. Much of the Cherry Street property is devoted to shipping and publication storage space which accounts for the smaller proportion of office space in this building:

	Office Floors, sq ft	Storage Area, sq ft	Balance,[a] sq ft	Total, sq ft
Race St	6324	1663	7459	15 446
Cherry St	3590	6800	2259	12 649
Total	9914	8463	9718	28 095
Arch St				14 486
				42 581

---

[a] Lobbies, hallways, stairways, toilets, rest rooms, elevators, walls, etc.



Block Plan Showing Location of ASTM Headquarters Properties.

Technical Activities

THERE are several indices of ASTM growth—number of new standards, technical publications issued, research projects started, and especially new and expanded technical committees. Only by a review of the minutes of the hundreds of committee meetings and by perusing the annual reports of these committees totaling 600 to 800 pages can one grasp the extent of the work. The ASTM BULLETIN *constantly highlights committee work. In the Annual Directors' Report reference can be made only to major new work and significant changes in committee scopes.*

Revised Committee Scopes and Jurisdictions:

Clay Pipe.—To permit the writing of specifications for factory-made joints, the scope of Committee C-4 on Clay Pipe has been modified to read as follows by the addition of the words in italics:

> "The formulation of specifications, nomenclature and methods of test for *vitrified* clay pipe, *vitrified clay liner plates, and factory-made compression joints* used for the conveyance of sewage, industrial waste, and storm waters; and for clay flue linings."

Electrical Insulating Materials.—Following the elimination from the jurisdiction of Committee D-9 of work involving liquids and gases to be covered by the new Committee D-27, a new scope of Committee D-9 has been approved as follows:

> "(1) Development of methods of sampling and testing, and formulation of specifications for solid electrical insulating materials, insulating varnishes, and any other insulating materials which are solid in normal use even though they may be applied in a fluid phase. Materials comprise both those designed as electrical insulation and those grades of other dielectric solids designed with controlled dielectric, piezoelectric, or magnetostrictive properties.
>
> "(2) Research, comprising studies of fundamental properties and application problems, on electrical insulating solids."

Metallic Materials for Electrical Heating, Electrical Resistance and Electrical Contacts.—While no action is being taken currently on a proposed revised program for Committee B-4 which at present limits its activities to metallic materials, there have been proposals that it cover such materials as silicon carbide and molybdenum disilicide which would require a change in the committee's title and scope. This might involve reclassifying the committee from the "B" group of committees to the "F" group which cover materials for specific applications. This matter is being studied further in Committee B-4.

Halogenated Organic Solvents and Solvent Mixtures.—As a result of its organization at the Boston Annual Meeting, 1958, a new Committee D-26 on Halogenated Organic Solvents and Solvent Mixtures adopted the following scope which has received Board approval:

> "The promotion of knowledge pertaining to halogenated organic solvents and admixtures thereof including formulation of specifications, definitions, and methods of test.
>
> "Standards peculiar to electrical insulating liquids, paint thinners and nonhalogenated components of admixtures normally are excluded from the scope of Committee D-26. Developments in these and other fields incidental to the work of Committee D-26 will be coordinated with the appropriate technical committees of the Society."

Major New or Suggested Activities:

Electrical Insulating Liquids and Gases.—The steadily growing interest in the field of liquids and gases used for electrical insulation and the fact that the former Subcommittee IV of Committee D-9 was virtually an entity in itself led to the recommendation that a new technical committee be established. This was approved by the Board. This new Committee D-27 on Electrical Insulating Liquids and Gases has the following scope:

> "The promotion of knowledge pertaining to electrical insulating liquids and gases, whether of synthetic or natural origin, and the recommendation of standards pertinent to these materials.
>
> "Standards peculiar to solid insulating materials and varnishes, and the development of standards pertaining to the nonelectrical uses of liquid and gaseous materials are excluded from the scope of Committee D-27. Developments in these fields incidental to the normal work of Committee D-27 will be coordinated with the appropriate technical committees of the Society."

Caulking and Sealing Compounds.—At its May, 1959 meeting, the Board approved the organization of a new technical committee to cover elastometric caulking and sealing compounds. There is intensive interest in polysulfite base type compounds and other types of sealants. These are widely used essentially as construction materials as distinct from the use of oil pigment types of caulking compounds. Committee D-1 on Paint, Varnish, Lacquer, and Related Products has a Subcommittee XIX on Putty and Related Compounds. The work done here and in the new technical committee will be coordinated.

Ore Sampling and Analysis.—With a number of organizations willing to participate in the development of information and test methods which might use statistical means for the sampling of bulk materials, including ores, a conference is being held on June 2 to consider specific needs and ascertain whether a new technical committee should be established. This would relate not only to the item of particular current interest —manganese ores—but to others as well, and could include fluxes and similar materials.

Graphite.—A working group to consider standards for graphite for nuclear use has been approved; the group will be appointed shortly and a meeting held. This has resulted from needs for prompt action in this area. The American Nuclear Society will cooperate, and is desirous that work be undertaken promptly. There are other uses for graphite and, depending upon the discussion and progress in the present working group, a new permanent technical committee may be established within the year.

Pigments.—There are many pigments for a variety of uses other than in paints and preservative coating. While some materials such as carbon black which may be used as pigments are the subject of other committee work (Committee D-24 on Carbon Black was organized in 1956), there is need for a conference to consider research and standardization work on other pigments. The Board has approved this activity.

Evaluating Airport and Highway Pavements and Surfaces—Last fall, the first International Conference on Skid Resistance was held at the University of Virginia with ASTM President K. B. Woods and many other ASTM members participating. There was apparent considerable interest in whether standards for skid resistance should be established, and as a result of discussion there is to be organized a joint committee to function in this field. The committee would be concerned with methods for evaluating airport and highway pavements and pavement surfaces, and the scope may include work on skid resistance, road roughness, methods of measuring deflection (Benkleman Beam, for instance), visibility, icing over bridges, etc. Membership would include representatives of existing technical committees which would have specific interest, including C-9 on Concrete and Concrete Aggregates, D-4 on Road and Paving Materials, and D-11 on Rubber and Rubber-Like Materials. There may be others.

Gaskets,—A Steering Committee appointed in 1958 has been discussing the need for expanded work in the field of gaskets of all kinds in addition to the activities in Committee D-11 on Rubber and Rubber-Like Materials. A recommendation is expected shortly from the Steering Committee.

Metallic Sheet Materials.—In response to a request from the Department of Defense a special committee has been established to develop adequate methods of evaluating metallic sheet materials, including their use in casings for solid fuels. This is a complicated matter since it involves multi-axial stresses and other special conditions. Several experts in this field are participating in the work, and several meetings have been held.

Ceramics for Electronic Uses.—With intensifying interest in the field of ceramic materials used in the electronics field, such as ferroelectric, ferromagnetic, and semiconducting ceramics, high-frequency ceramic insulation, and so on, there is the possibility that some standardization activities should be developed, and perhaps centered in a single technical committee. There are at least five ASTM committees whose work is related to the electrical ceramic areas, these being Committees A-6, B-9, C-21, D-9, and F-1. Accordingly a conference of interested parties, including representation from the American Ceramic Society, is being arranged to establish a consensus on certain standardization problems. It is of interest that the American Ceramic Society, which has cooperated very closely in many facets of ASTM work, has established an Electronics Division headed by John H. Koenig of Rutgers University.

General Problems.—Health Hazards.—In approving a special wording to be inserted in a new specification for leaded steels where it is desirable that there be adequate ventilation when manufacturing involves elevated temperatures, the Board reemphasized its previous policy of requesting technical committees to incorporate information where potential health hazards may be involved. Advice to this effect appears in the records and the Manual for Technical Committee Officers, but now is extended specifically to include specifications as well as test methods. It was noted that a number of organizations present as much information as possible, partly in order to prevent legal problems.

Various Types of Methods.—Already there are a variety of test methods such as research methods, field methods, and so on, which have been published as ASTM standards on the recommendation of various committees. The Board has stated that there need be no limitations put on the type of test published by the Society other than to point out very clearly in the scope the application of the methods.

Packing, Marking and Loading Methods.—Should requirements on packing, marking, shipping, and preservation be a part of ASTM specifications? This matter has been discussed in several of the technical committees and in the

Administrative Committee on Standards, and was reviewed recently by the Board. The Board believes that the recommendation on packing, marking, and so on embodied in Section 12 of the Proposed Recommendations on the Form of ASTM Specifications adequately covered this subject. These recommendations are printed below. (A copy of the complete document is available from ASTM Headquarters without charge on request.)

A number of industries through their trade groups or in other ways have established very satisfactory recommendations concerning packing and shipping of numerous products. It is frequently considered sufficient to refer to these recommendations even though there may be no requirement in the ASTM specifications.

From the Proposed Recommendations on Form of Specifications:

Packing, Marking, Shipping, and Preservation

12. Where it is customary to package, box, crate, wrap, or otherwise protect the material or product for shipment, the general requirements for such action shall be stated. If it is necessary to protect the material from damage in ordinary handling or from moisture or heat, etc., the general nature of the wrapping or packing should be stated. If the material is to be shipped in packages of definite sizes these should be specified. It is customary to state the nature of the information to be marked or included on the package or on a label or tag attached thereto. This usually includes such details as the name and brand or trade mark of the manufacturer, type of material, size, date of manufacture, and ASTM designation of the specification to which the material conforms. It is sometimes necessary to state the location of the marking on the article, such as on pipe, tubes, sheet, plate and shapes.

In essence the Board confirms that it is entirely appropriate when so decided in the technical committee for ASTM standards to include packing and shipping requirements.

Psychophysical (Human Aspects) Testing.—Although the Board of Directors has been quite willing to consider specific activities in the field of ultimate consumer goods, in which area an administrative committee functioned for some time, it was difficult to reach any conclusions on tangible projects. This committee then served more or less as a stand-by committee to handle any technical problems which might arise. Two years ago the concept of the committee was changed somewhat and its title revised to cover "end-use products." In January the Board decided, with the acquiescence of the committee, to discharge it with thanks.

Concurrently approval was given to a conference of interested members to evaluate the organization of a technical committee which would deal with the so-called human aspects of testing, termed psychophysical testing or organoleptic aspects. This is an area of concern to a number of industries and some tangible program of work might be developed.

National and Other Meetings

MEETINGS provide the personal area for the exchange of ideas—a must for continued progress in research and standards for materials.

1958 Annual Meeting:

The Sixty-first Annual Meeting of ASTM in Boston, June 22 to 27, 1958, was by all standards the most successful in the history of the Society. Nearly 900 technical committee meetings were held.

In addition, a significant program of more than 40 technical sessions and numerous special luncheons and dinners were on the agenda. The committee reports presented are to be found in the 1958 *Proceedings* as well as some technical papers. Other papers have appeared in various issues of the ASTM BULLETIN and in separate special technical publications (STP's). A condensed review of the meeting was featured in the July 1958 ASTM BULLETIN. The outstanding entertainment program for the ladies, developed by the General Committee on Arrangements, attracted some 550 of the members' families. The special dinner and ASTM Boston Pops Concert in Symphony Hall drew over 2200.

1959 Committee Week:

Committee Week, held this year in Pittsburgh the week of February 2, provided technical committee members with the opportunity to meet, discuss, and work on their problems. It has been found most convenient, particularly for technical committee people who serve on more than one committee, to hold coordinated meetings, rather than to hold the committee meetings separately. Nearly 300 committee meetings were held.

Two industry luncheons—for the Metals Industry, and for the Building Materials Industry—were held. These, as well as the outstanding dinner, were described in the April 1959 BULLETIN.

District Support of National Meetings:

The New England District through its Annual Meeting arrangements committee performed yeoman service for the Society at the 1958 Annual Meeting. Excellent cooperation with the press was secured; ladies' entertainment was arranged; a dinner and a concert by the Boston Pops orchestra was sponsored and will long be remembered as the entertainment highlight of an outstanding meeting. In addition to the President's Luncheon, four industry luncheons were held, with prominent speakers covering the Instrument and Apparatus Industry; Copper and Brass; Road Materials; and Petroleum.

Future Meetings:

Annual Meetings of the Society have been scheduled as follows:

June 26–July 1, 1960—Atlantic City, N. J.
June 25–30, 1961—Atlantic City, N. J.
June 24–29, 1962—New York, N. Y.
June 23–28, 1963—Atlantic City, N. J.
June 21–26, 1964—Chicago, Ill.

Committee Week and Spring Meetings are as follows:

February 1–5, 1960—Chicago, Ill.
January 29–February 3, 1961—Cincinnati, Ohio
February 5–9, 1962—Dallas, Tex.
February 3–8, 1963—Montreal, Quebec, Canada

Pacific Area National Meetings are scheduled as shown:

October 11–16, 1959—San Francisco, Calif.
September 30–October 5, 1962—Los Angeles, Calif.

Pacific Area National Meeting.—The Third Pacific Area National Meeting in San Francisco in October will have the most extensive technical program of any ASTM meeting. There are at least 52 technical sessions, which means that for most days there will be six sessions running simultaneously. In addition there are the President's National Luncheon, five industry luncheons, an outstanding program of entertainment, largely for the ladies, and a list of industrial trips from which members can make a selection. The General Committee on Arrangements for this meeting includes many leading members and committee members on the West

Coast. This group has and will continue to work aggressively to make this meeting outstanding in all respects.

A special booklet listing all technical papers and features of the meeting will be sent to the members late in May.

District and Related Activities

ASTM has seventeen districts serving members throughout the nation with the exception of the Northwest and part of the Midwest. These districts, administered by local councils, provide members with the opportunity to hear outstanding technical talks, to meet with national officers, and to take part in ASTM activities at both the local and national level.

During the year the activities of the districts were maintained at a high level. Two new districts were organized. The Central New York District was formed from parts of the Western New York–Ontario and New York Districts, taking into consideration the concentration of industry in the Mohawk Valley. The inaugural meeting was held in Troy, N. Y. The Rocky Mountain District, comprised of Arizona, New Mexico, Utah, Colorado, Wyoming, and a small part of Texas, was formed in view of the rapid industrialization of the area. A full-day session on "Explosives and Their Relationship to Materials" was held at Salt Lake City as part of the inaugural ceremonies.

Student Membership Awards were presented by twelve districts—a new high. In addition, another district is studying plans to include a program. Some 750 awards were made by districts or by the Board of Directors.

President Woods spoke before 14 districts, making some 30 appearances in all. Many of these meetings were at universities and colleges and were jointly sponsored. Vice-Presidents Bates and LaQue each addressed two meetings. Every district held at least one meeting during the year.

The accompanying listing shows the date and place of each district meeting. In addition, two special meetings were held outside districts. The vast majority of the meetings were jointly sponsored or were held in cooperation with another technical or educational group. Complete news accounts of the meetings were reported throughout the year in the ASTM BULLETIN.

District Meetings:

Southeast District (jointly with ASCE)	October 6, 1958	University of Florida, Gainesville, Fla.
Southeast District (jointly with ASCE, Ga. Engr. Soc., SAME, ASME)	October 8, 1958	Georgia Institute of Technology, Atlanta, Ga.
Southeast District (jointly with ASCE, Tenn. Soc. of Prof. Engrs., SAME)	October 9, 1958	University of Tennessee, Knoxville, Tenn.
St. Louis District (jointly with AWS)	October 9, 1958	Washington University, St. Louis, Mo.
Ohio Valley District (jointly with Ky. Soc. Prof. Engrs.)	October 10, 1958	University of Kentucky, Lexington, Ky.
Philadelphia District (jointly with ASTM Committee F-1)	October 13, 1958	Philadelphia, Pa.
Chicago District (jointly with ASTM Committee D-10)	October 16, 1958	Madison, Wis.

New England District (jointly with ASCE, Boston Soc. of Civil Engrs.)	October 30, 1958	Cambridge, Mass.
Northern California District (jointly with ASME, AIEE)	November 3, 1958	San Francisco, Calif.
Detroit District (jointly with Detroit Paint, Varnish & Lacquer Assn.)	November 10, 1958	Detroit, Mich.
Pittsburgh District (jointly with AIEE)	November 11, 1958	Pittsburgh, Pa.
Central New York District (jointly with ASCE)	November 17, 1958	Rensselaer Polytechnic Inst., Troy, N. Y.
Washington District	November 19, 1958	Washington, D. C.
Philadelphia District (jointly with ASCE)	November 20, 1958	Villanova University, Villanova, Pa.
Chicago District (jointly with ASTM Committees C-1 and C-9)	December 3, 1958	Purdue University, Lafayette, Ind.
New York District (jointly with AIEE, ASCE)	January 13, 1959	New York City
Chicago District (jointly with ASTM Committee A-1)	January 14, 1959	Chicago, Ill.
Washington District	January 21, 1959	Raleigh, N. C.
Philadelphia District (jointly with Franklin Institute)	February 25, 1959	Philadelphia, Pa.
Southwest District (jointly with ASCE)	February 26, 1959	Rice Institute, Houston, Tex.
Southwest District (jointly with Texas A & M)	March 2, 1959	Texas A & M College, College Station, Tex.
Southwest District (jointly with ASCE and CSI)	March 3, 1959	Southern Methodist University, Dallas, Tex.
Southwest District (jointly with University of Oklahoma)	March 4, 1959	Norman, Okla.
Rocky Mountain District (jointly with University of Utah)	March 6, 1959	University of Utah, Salt Lake City, Utah
Southern California District (jointly with Am. Concrete Institute)	March 9, 1959	Los Angeles, Calif.
Northern California District (jointly with ASCE)	March 10, 1959	San Francisco, Calif.
Special Meeting (jointly with University of Washington)	March 13, 1959	Seattle, Wash.
Special Meeting (jointly with ASM, ACS, ANS, ISA, AIEE, ASCE, IRE, ASME, Washington State Soc. of Prof. Engrs.)	March 17, 1959	Richland, Wash.
Chicago District	March 23, 1959	Chicago, Ill.
Cleveland District (jointly with NACE, ASM)	March 31, 1959	Cleveland, Ohio
Chicago District (jointly with Iowa State College)	April 14, 1959	Ames, Iowa
New England District (jointly with ASCE)	April 18, 1959	University of Dartmouth, Hanover, N. H.
New York District	April 23, 1959	New York City
Central New York District (jointly with ASM)	April 23, 1959	Utica, N. Y.
Detroit District	April 29, 1959	Detroit, Mich.
Pittsburgh District	April 30, 1959	Pittsburgh, Pa.
Western New York – Ontario District	May 8, 1959	Rochester Institute of Technology, Rochester, N. Y.

The New England and Pittsburgh Districts were active as hosts for the 1958 Annual Meeting and 1959 Committee Week Meeting, respectively. A more complete report is found under "National Meetings."

Districts were active not only in organizing technical sessions, but also in local technical councils, Science Fairs, Engineering Week Celebrations, and in assisting the Society's Advisory Committee on Corrosion.

The Administrative Committee on District Activities held a meeting during the 1958 Annual Meeting and sponsored a breakfast to which District Council Officers were invited. Once again, this proved an excellent opportunity for the exchange of ideas relating to the promotion of the Society and its activities.

Membership

WE are obviously concerned with membership, not only to insure the Society's growth numerically but also along technical lines. More companies and individuals must be acquainted with our work and asked to participate.

On May 1, 1959, the membership of the Society totaled 9786, compared with a total of 9359 one year ago. Gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	May 1, 1958	May 1, 1959	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	29	26	...	...	3	...	...	...	3	...	3[a]
Perpetuity and Life	13	13	...	...	1	...	...	1	1	1	...
Sustaining	417	406	...	...	...	12	...	1	12	1	11[a]
Company, Firm, etc.	2041	2076	79	15	...	17	17	129	111	146	35
Individual, etc.	6603	6961	318	124	52	6	53	805	500	858	358
Associate	256	304	13	11	...	35	...	107	59	107	48
Total	9359	9786	410	150	56	70	70	1043	686	1113	427
Student	1031	1484	64	544	...	46	...	1107	654	1107	453

[a] Net loss.

It is interesting to compare our present membership of 9786 with the figures for ten and twenty years ago; in 1949 our total membership was 6620, and in 1939 it was 4199.

In 1958 there were more new members than in the previous high year, 1956—1024 compared to 1014. The average in several previous years was from 602 to 775. Our net gain in 1958 was 427.

Associate and Student Memberships:

The class of Associate Membership, which extends to 30 years of age and replaced the former junior membership with its 27-year age limit, continues to grow as noted in the table.

Because of the influence of student membership prize awards, which recognize outstanding students in courses related to the Society's work, the very encouraging growth in this class has continued. The Society itself and many of the districts, in addition to individuals and companies, are sponsoring student membership awards.

Sustaining Members:

There has been one new sustaining member in the period from May, 1958, to May, 1959, and 12 transfers to company memberships. The new sustaining member is:

Phoenix Cement Co., Division of American Cement Corp.

50-Year Members:

There are two individuals and twelve organizations as noted below that this year have been connected with the Society continuously for 50 years:

Everett W. Boughton
Joseph Brobston

Bird and Son, Inc.
Commonwealth Edison Co.
Electrical Testing Laboratories, Inc.
Heppenstall Co.
Midvale-Heppenstall Co.
Phelps Dodge Copper Products Corp.
Raymond Concrete Pile Co.
Schenectady Varnish Co., Inc.
The Sherwin-Williams Co.
Tulane University of Louisiana
Western Electric Co., Inc., Hawthorne Works
Westinghouse Air Brake Co.

These members will be recognized at the Annual Meeting.

40-Year Members:

There are 44 members who this year have completed 40 years of membership, bringing the total number of certificates issued to the "Forty-Year Club" to 462. The members who will receive the 40-Year Certificates at this Annual Meeting are as follows:

Arizona State Highway Dept.
The Baltimore & Ohio Railroad Co.
Ray T. Bayless
Robert D. Bonney
W. H. Campen
Canada Cement Co., Ltd.
The Chesapeake & Ohio Railway Co.
City and Guilds College Library
The Colorado Fuel and Iron Corp., Wickwire Spencer Steel Div., Claymont Plant
Columbia-Geneva Steel Div., U. S. Steel Corp.
T. G. Delbridge
Edgar Hutton Dix, Jr.
Driver-Harris Co.
E. I. du Pont de Nemours and Co., Inc.
Erie Railroad Co.
Georgia Institute of Technology Library
Georgia, State Highway Department of
Max Herzog
Francis M. Howell
Kansas State Highway Commission
Kellogg Switchboard and Supply Co.
Lone Star Cement Corp.
Charles H. Marshall
F. R. McMillan
Montana State Highway Dept.
The National Supply Co., Tubular Div.
New York Central System
New York, New Haven & Hartford Railroad Co.
Northern States Power Co.
Phoenix Bridge Co.
Lucius Pitkin, Inc.
Rochester, City of, Department of Public Works, Division of Engineering
Nathan C. Rockwood
St. Joseph Lead Co.
The Shawinigan Engineering Co., Ltd.
The Singer Manufacturing Co.
A. O. Smith Corp.
Union Pacific Railroad Co.
R. T. Vanderbilt Co., Inc.
Stephen F. Voorhees
Walter M. Weil
Wellington Sears Co., Inc.
West Coast Lumbermen's Assn.
Guy M. Williams

Deaths:

The Society has lost the following 55 members through death (figures in parentheses are dates of membership):

Charles W. Allen (1940)
Eugene B. Allen (1947)
F. E. Amthor (1926)
Preston F. Bailey (1957)
Edward Bartow (1932)
H. C. Berry (1905)
Robert F. Blanks (1932)
Robinson D. Buck (1947)
John J. Chapman (1955)
Burr O. Collins (1936)
V. A. Crosby (1934)
Merton H. Davey (1957)
W. H. Eisenman (1944)
Luigi Emanueli (1930)
Stanley H. Franklin (1946)
Walter H. Fulweiler (1909)
Samuel A. Gordon (1949)
Samuel S. Gutkin (1953)
Frank E. Harley (1958)
Fred L. Hastings (1957)
Walter A. Heinrich (1932)
Max G. J. Hoffmann (1957)
Wesley Winans Horner (1936)
Charles B. Karlson (1929)
A. J. Kearfott (1957)
C. F. Kettering (1915)
C. E. Lamarche (1944)

Clarence Lamoreaux (1949)
C. P. Lewellen (1939)
William T. Long (1953)
Donald S. MacBride (1930)
James T. MacKenzie (1918)
Joseph J. Markey (1943)
John L. McCloud (1924)
John C. McRae (1947)
Kenneth V. Merrill (1940)
William Morrow (1940)
C. A. Neusbaum (1953)
Frank J. O'Neil (1946)
R. O. Peterson (1951)
Howell L. Phillips (1954)
L. E. Pitzer (1946)
Bolla E. Pollard (1949)
Donald E. Sharp (1946)
B. G. Simek (1949)
E. A. Snyder (1925)
Harry Z. Snyder (1957)
Leon Sternberg (1953)
A. E. Stoddard (1922)
Henry L. Taylor (1953)
Tsukumo Tomonari (1950)
C. P. Van Gundy (1903)
Arthur R. Weismantle (1949)
Willard H. Woodstock (1957)
William W. Wright (1956)

In addition to the above, the following representatives of company members of the Society passed away:

Warren Ames, B. C. Ames Co.
R. R. Bowser, Bowser-Morner Testing Laboratories
Paul J. DeKoning, Michigan State University, Department of Applied Mechanics
Page Fisk, City of Long Beach, California, Chemical and Testing Lab.
Frank P. Gilligan, The Henry Souther Engineering Co.
Chester Hacking, William H. Haskell Manufacturing Co.
Chester C. Kelsey, Asbestos-Cement Products Assn.
Martin S. Maier, U. S. Asbestos Division of Raybestos-Manhattan, Inc.
Frederick A. McCoy, Sheffield Steel, Division of Armco Steel Corp.
A. D. Schultz, Eaton Stamping Div., Eaton Manufacturing Co.
George J. Wingate, Jersey Testing Laboratories

A number of these men had been affiliated with the Society for many years and rendered outstanding services, particularly through work on the technical committees. The various issues of the ASTM BULLETIN have included appropriate notices and appreciation of their work.

Honors and Awards

THROUGH election to national office and honorary membership, giving awards of merit, and recognizing outstanding technical contributions, ASTM each year honors men who have given outstanding service to the Society and its work.

Descriptions of the various awards as well as the personnel of the lecture and award committees are given in the ASTM Year Book.

Recipients of honors and awards to be given at the 1959 Annual Meeting are indicated below:

Honorary Membership:

Douglas E. Parsons, Chief, Building Technology Division, National Bureau of Standards.

Jerome Strauss, Consultant (retired Vice-President, Vanadium Corporation of America).

Award of Merit:

Victor E. Grotlisch
George O. Hiers
Elmer G. Kimmich
Bryant Mather
Mary R. Norton
Lewis S. Reid
Claude K. Rice
Henry J. Schweim
Benoit J. Sirois
Herbert W. Stuart
Charles K. Strobel
Beaumont Thomas
George L. Werley

Recognition of Technical Papers:

Charles B. Dudley Medal (research).—R. J. MacDonald, R. L. Carlson, and

W. T. Lankford for their paper "Effects of Strain Rate and Temperature on the Stress-Strain Relations of Deep-Drawing Steel."

Richard L. Templin Award (testing).—J. H. Westbrook for his paper "Microhardness Testing at High Temperatures."

Sam Tour Award (corrosion).—J. B. Rittenhouse for his paper "The Corrosion and Ignition of Titanium in Fuming Nitric Acid."

Sanford E. Thompson Award (concrete, sponsored by Committee C-9).—George Verbeck for his paper "Carbonation of Hydrated Portland Cement."

C. A. Hogentogler Award (soils), sponsored by Committee D-18).—W. E. Schmid for his paper "The Permeability of Soils and the Concept of a Stationary Boundary-Layer."

Recognition of Outstanding Contributions:

Max Hecht Award (industrial water, sponsored by Committee D-19).—L. D. Betz, General Manager, Betz Laboratories, Inc., Philadelphia, Pa.

Committee D-12 Award (soaps and other detergents).—J. C. Harris, Director, Application Research Dept., Monsanto Chemical Co., Dayton, Ohio.

Harold DeWitt Smith Memorial Award (textiles).—W. J. Hamburger, Director, Fabric Research Laboratories, Inc., Dedham, Mass.

ASTM Lectures:

The *Edgar Marburg Lecture* on "New Polymers—New Problems" will be presented on Tuesday afternoon, June 23, at Haddon Hall, Atlantic City, by Dr. Herman F. Mark, Director, Polymer Research Institute, Polytechnic Institute of Brooklyn.

The *H. W. Gillett Memorial Lecture* on "The Role of Dislocations in Plastic Deformation (of Metals)" will be presented on Wednesday afternoon, June 24, also at Haddon Hall, Atlantic City, by Dr. John C. Fisher, Physicist, Metallurgy & Ceramics, Research Department, General Electric Co.

Finances

THE Finance Committee recognizes the importance of sound finances in underwriting much of the constantly expanding work. The Society's financial condition has for years been relatively healthy. This is attributable to the excellent support received from industry and also to the value of ASTM service—publications, standards, research data.

Report for 1958:

Full details of the fiscal operations of the Society for 1958 are included in the report of the auditors appearing in Appendix I.[1]

Operating Receipts:

The estimated receipts for December 1, 1957, through November 30, 1958 of $1,065,000 actually developed to be $1,084,016. Of this amount, receipts from dues and entrance fees were $297,801 or 27.5 per cent; receipts from sales of publications were $657,020 or 60.6 per cent; and receipts from miscellaneous sources consisting principally of advertising, income from investments, exhibits and registration fees totaled $129,195 or 11.9 per cent. These percentages do not differ markedly from the previous year.

Receipts from membership fees still

---

[1] See p. 81.

continue to be under 30 per cent of the total.

Operating Disbursements:

Total disbursements were $1,252,413 which included a reserve of $118,000 to complete payment for the 1958 Book of Standards and the 1958 *Proceedings*.

A total of $577,036 was spent for publications, which compares with $431,000 for the previous year. Payments for the 1958 Book of Standards were the largest item by far. Final costs for the Book will be handled in 1959.

Expenditures for salaries, which included extra summer help and considerable overtime, reached $406,126. This figure represents 32.4 per cent of total disbursements. Cost of employee benefits, including the major medical policy and covering new participants in the retirement plan, totaled $48,391.

Operating Deficit.—Thus for 1958 the financial operations showed a deficit of about $168,000. This was considerably higher than budgeted, but there was no curtailment in publication or other activities. Too, it should be remembered that this was the third in our three-year period of the Book of Standards which normally is the low of the three years for receipts.

Balance Sheet:

On November 27 the total assets in General Funds were $996,800 compared with $1,017,100 a year ago. In all funds—General, Building, Medal, etc.—total assets were $1,676,800 as compared with $1,663,500 a year ago. It should be pointed out that in the Balance Sheet no account has been taken of the assets of the Society in the form of publications in stock. This is consistent with many years' practice in this respect and, of course, results in a more conservative statement. An inventory of the principal technical publications as of May 1, 1959, is summarized below:

Proceedings (All back copies from 1905 to 1957 incl)	2 780
1958 Book of ASTM Standards:	
Part 1	10 772
Part 2	10 287
Part 3	10 679
Part 4	9 858
Part 5	8 442
Part 6	5 847
Part 7	5 603
Part 8	8 228
Part 9	8 843
Part 10	6 849
ASTM Methods of Chemical Analysis	3 335
Special Compilations of Standards	38 373
Selected Standards for Use in College Curricula	11 249
Symposiums, STP's, and Special Reprints	202 104
Reprints of Standards (approx.)	265 000
Radiographic Standards for Steel Castings	100
Radiographic Standards for Aluminum and Magnesium Castings	13
Radiography of Steel Welds	29

Not Included in Inventory:

(1) Viscosity Temperature Charts
(2) Hardenability Charts
(3) Manual of Engine Test Methods and Supplements
(4) Marburg Lectures
(5) Cotton Yarn Boards
(6) Petroleum Charts

Building Fund:

Assets in the Building Fund as of November, 1958, which include the current Race Street Headquarters, the Cherry Street Property, and the balance of the lot immediately adjoining this, total $313,300.

The loan from ASTM General Funds which enabled the procurement of the Cherry Street property amounted to $29,325 as of November 27, 1958. It has been amortized in the amount of $8700 yearly, but with the acquisition of the Arch Street property in January, 1959, this is being changed. This latter property cost about $130,000 of which General Funds advanced $125,000. The new note, now $154,325, is to be amortized at $17,000 yearly. These monies will come from allocations to Headquarters occupancy costs, a part of the operating budget.

Investments:

In the operation of the Investment Fund, the Society had continuing advice from our investment counsel—Loomis, Sayles & Co. This Fund was set up in 1956 by transferring to it assets from the various operating funds such as General, Building, Research, etc., with these funds owning *pro rata* a share in the Investment Fund. The operating funds proportionately benefit from income and growth of the Fund.

During the year a number of changes in investments were made. These are detailed in the Auditors' Report which is appended.[1] An important decision has been made gradually to liquidate our Mutual Funds; they will thus come under the guidance of investment counsel. We are keeping equities in that area of 50 to 55 per cent.

As of November 27, 1958, the book value or cost of the securities in the fund totaled about $802,020; market value was $1,011,800. Total income was $39,800. The Society's holdings of mutual funds are included in the above totals of the Investment Fund. As of November 27, Mutual holdings had a market value of about $170,350.

1959 Budget:

From the accompanying summary of the 1959 budget, which as usual is conservatively estimated for income and liberal for costs, it will be seen that a small favorable balance is anticipated for the current year. It is hoped that the increased dues for Company and Sustaining Members and larger income from sales of publications will create the substantial favorable balance we should have here this year.

BUDGET SUMMARY—1959.

		Per cent
Estimated Receipts:		
Dues and Entrance Fees	$380 175	28.2
Publications	857 500	63.6
Miscellaneous	110 825	8.2
TOTAL CURRENT RECEIPTS	1 348 500	100.0
Estimated Disbursements:		
Publications	559 500	42.4
Salaries	435 000	33.0
General Office Expenses	112 500	8.5
Technical Committee Expenses	16 000	1.2
Meetings	38 350	2.9
Headquarters Occupancy Expenses	60 000	4.5
Retirement, Insurance Expenses	53 500	4.1
Miscellaneous	45 250	3.4
TOTAL OPERATING DISBURSEMENTS	1 320 100	100.0
ESTIMATED GAIN	28 400	
TOTAL	$1 348 500	

Headquarters Staff:

As the technical, editorial, and other work which the Staff handles continues to grow, it is necessary to secure additional competent personnel. Each new technical committee requires some time of a member of the Technical Staff since someone at Headquarters follows closely the work of each main committee

On April 13, Robert A. Lukens, who has a Masters Degree in Chemical Engineering from Rensselaer Polytechnic Institute, and who has had considerable experience in chemical plant design and construction and the use of specifications, became a member of the Technical Staff. Tentatively he is assigned to standards editorial work and is reporting to the Technical Secretary and through him to the Executive Secretary.

Better to recognize responsibilities

in connection with the ASTM BULLETIN, effective June 1, Frank Y. Speight, Assistant Technical Secretary, will also be designated as Features Editor and A. Q. Mowbray becomes Assistant Editor. Mr. Mowbray has been concentrating on the BULLETIN in recent months.

Cooperative Activities

EVERY ASTM project, whether in research or standards or meetings, is cooperative in the sense that the consumers and producers cooperate to carry out the work. Many other societies and associations take part either through sponsorship of joint committees, handling of technical committee secretarial work, or in other ways. Thus cooperation is a key word in ASTM.

As noted in the May BULLETIN, there seems to be a trend toward having more technical work sponsored jointly, with ASTM and other technical or professional groups closely cooperating. In some areas joint committees have functioned for over 30 years, as in the case of the effect of temperature on the properties of metals. Other notable examples include filler metal (welding), soaps and detergents, automotive rubber, plastic pipe, cellulose, and leather.

Another extremely important area of cooperation involves the official representation on technical committees by upwards of 200 societies, trade associations, and technical organizations. The secretariat of some of our most important technical committees is handled by individuals on the staffs of trade associations. This is a tremendous service not only to their members but to the respective industries generally.

Only a few activities during the year and some contemplated have been singled out for reference.

ASTM was an endorser of the National Conference on the Application of Electrical Insulation held in Cleveland under the sponsorship of the AIEE. There was an ASTM booth in the special exhibit.

Because of our interest in documentation and publication activities, the Society was represented at Western Reserve University's Council on Documentation Research last year. Dr. F. T. Sisco, Director of Engineering Foundation, is continuing to represent ASTM on that council.

We were glad to cooperate with the AIME for its Metallurgical Society Conference on Stress-Corrosion Fracture held as an intersociety venture in Pittsburgh in April. Outstanding authorities participated. In this connection it is of interest that demand still continues for the very extensive Symposium on Stress-Corrosion Cracking of Metals comprising some 30 technical papers held jointly with AIME almost fifteen years ago. In this same area, a publication will probably be forthcoming in the next few months as a result of a recent questionnaire on problems involving stress-corrosion cracking of austenitic steels, with NACE and ASTM cooperating.

The President and Executive Secretary attended a meeting of the Pan-American Society of Engineers in Montreal in September, the President being an honorary United States delegate and the Executive Secretary serving as a representative of the ASME. The Executive Secretary also serves similarly on the Committee on International Relations of the Engineers Joint Council.

The Society took part in an extensive

symposium on the National and International Aspects of Systems of Unit held as part of the December meeting of Section M, Engineering, of the American Association for the Advancement of Science. Officers of Committee E-1 on Methods of Testing presented a paper and the Executive Secretary cooperated in developing the symposium. Reference has been made to this in the April BULLETIN in a short article "Units of Measurement—Babel?"

Respectfully submitted on behalf of the Board of Directors,

K. B. WOODS,
President.

R. J. PAINTER,
Executive Secretary.

June, 1959

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR NOVEMBER 27, 1957 TO NOVEMBER 28, 1958

Philadelphia, December 12, 1958

MR. R. J. PAINTER, *Executive Secretary*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pa.

Dear Sir:

We have examined the books and accounts of the American Society for Testing Materials for the period November 27, 1957 to November 28, 1958. We did not make a detailed audit of all transactions, but made tests to the extent we considered appropriate in determining the accuracy of the accounts.

Cash on deposit in checking and savings accounts and cash represented by certificates of deposit were verified by direct correspondence with the various depositories and reconciled with the cash records. The certificates of deposit were examined by us.

We verified by direct correspondence 100 accounts of Publications receivable and replies received disclosed only insignificant differences.

The distribution of income and the determination of participations in the principal of the investment fund were made as of November 17, 1958; therefore, all schedules and the market value of the securities of the investment fund are reflected as of November 17, 1958. The securities held by the investment fund, and securities held by other funds, were examined by us on December 12, 1958. Income from investments for the period under review was verified by us and found to be properly recorded on the books of the Society.

The employees' contributory pension plan was created on May 1, 1957, and the Treasury Department has approved the trust as a qualified trust under section 401(a) of the Internal Revenue Code. During the current fiscal year, the Society contributed $33,000 as their share of the costs of the plan. The right is reserved by the Society to amend or discontinue the plan.

Under date of November 3, 1958 the Society entered into an agreement for the purchase of premises 1921–23 Arch Street and 1920–22 Cherry Street, Philadelphia, Pennsylvania, for a consideration of $128,000. A deposit of $15,000 was made by the Society when the agreement was executed.

We have prepared and submit herewith balance sheet as of November 28, 1958, and statement of cash receipts and disbursements and operations for the period November 27, 1957 to November 28, 1958, and other supporting schedules which, in our opinion, present fairly the financial position of the Society at November 28, 1958 and the results of its operations for the period ended that date.

Respectfully submitted,
JOHN HEINS & Co

BALANCE SHEET AS OF NOVEMBER 28, 1958

(Including Special and Designated Funds)

ASSETS

General Funds:				
Current:				
Cash			$204 595.58	
Investment Fund Participation (at cost)			635 675.83	
Accounts Receivable			85 620.38	
Loans Receivable (Building Fund)			29 325.00	
Advance to Forest Products Laboratory			500.00	
Travel Deposit			425.00	
Prepaid Expenses			1 885.30	
Total Current Assets—General Funds			958 027.09	
Furniture and Fixtures (depreciated book value)			38 745.89	
Total Assets—General Funds				$ 996 772.98
Building Fund:				
Cash			1 202.43	
Investment Fund Participation (at cost)			30 401.04	
Deposit on Real Estate			15 000.00	
Land, Cost			68 459.42	
Buildings and Construction Costs		$282 083.47		
Less, Accumulated Depreciation		83 800.00	198 283.47	
Total Assets—Building Funds				313 346.36
Other Special and Designated Funds:				
Cash:				
ASTM Research Fund		13 220.88		
Dudley Medal and Marburg Lecture Fund	$193.95			
Richard L. Templin Award Fund	256.65			
Sam Tour Award Fund	382.69			
F. E. Richart Award Fund	90.54			
Total Medal and Lecture Funds		923.83		
Committee Funds		84 131.05	98 275.76	
Investment Fund Participation (at cost):				
ASTM Research Fund		83 155.13		
Dudley Medal and Marburg Lecture Fund		7 419.40		
Richard L. Templin Award Fund		2 374.22		
Sam Tour Award Fund		1 847.84		
F. E. Richart Award Fund		1 119.90		
Committee Funds		123 063.32	218 979.81	
Other Investments (at cost:)				
ASTM Research Fund		10 074.63		
Committee Funds		39 325.00	49 399.63	
Total Assets—Special and Designated Funds				366 655.20
Total Assets				$1 676 774.54

LIABILITIES

General Funds:			
Current:			
Loans Payable—Investment Fund		50 000.00	
Advance Collections		$20 857.58	
Accounts Payable:			
Joint Committee on X-Ray Diffraction		195 462.99	
Joint Symposium, Nuclear Meeting		1 476.77	
Pennsylvania Sales Tax		178.76	
Deferred Income		221.35	
Provision for:			
1958 Proceedings		39 000.00	
1958 Book of Standards		79 000.00	
Total Current Liabilities—General Funds		386 197.45	
Life Membership Fund	$4 017.94		
Publication Fund	16 841.80		
Book of Standards Reserve	161 030.02		
Executive Retirement Reserve	14 773.60		
Retirement Fund Reserve	37 044.04		
Reserve for Depreciation of Investments	20 000.00		
Reserve for Additional Cost of Replacement of Headquarters Building	35 000.00		
Special Annual Meeting Entertainment Account	4 661.64		
	293 369.04		
Surplus	317 206.49	610 575.53	
Total Liabilities and Surplus—General Funds			$ 996 772.98
Building Fund:			
Contributions from Members		188 912.65	
Contributions from ASTM General Fund		90 764.57	
Notes Payable—General Fund		29 325.00	
Accumulated Income, Net		4 344.14	
Total Building Fund			313 346.36
Other Special and Designated Funds:			
ASTM Research Fund:			
Principal	96 229.76		
Income	10 220.88	106 450.64	
Dudley Medal and Marburg Lecture Fund:			
Principal	7 419.40		
Income	193.95	7 613.35	
Richard L. Templin Award Fund:			
Principal	2 374.22		
Income	256.65	2 630.87	
Sam Tour Award Fund:			
Principal	1 847.84		
Income	382.69	2 230.53	
F. E. Richart Award Fund:			
Principal	1 119.90		
Income	90.54	1 210.44	
Committee Funds, Unexpended Balance		246 519.37	
Total Special and Designated Funds			366 655.20
Total Liabilities and Funds			$1 676 774.54

In Addition to the General Funds of the Society the Treasurer Had on Hand the Following Funds as of November 28, 1958

COMMITTEE FUNDS

Committee A-1	On steel	$1 072.28
Committee A-5	On corrosion of iron and steel	394.11
Committee B-3	On corrosion of non-ferrous metals and alloys	2 491.06
Committee B-4	On metallic materials for electrical heating, electrical resistance, and electrical contacts	62.36
Committee B-5	On copper and copper alloys, cast and wrought	21.49
Committee B-6	On die cast metals and alloys	3 406.87
Committee B-8	On electrodeposited metallic coatings	14.87
Committee C-1	On cementitious, ceramic, concrete, and masonry materials:	
	General funds	1 941.84
	Cement reference laboratory	244.14
	Cement reference laboratory—inspection	3 506.41
Committee C-9	On concrete and concrete aggregates	161.48
	Sanford E Thompson Medal Award	1 121.00
Committee C-14	On glass and glass products	49.22
Committee C-15	On manufactured masonry units	560.66
Committee C-16	On thermal insulating materials	132.00
Committee C-18	On natural building stones	458.95
Committee D-1	On paint, varnish, lacquer, and related products	66.39
Committee D-2	On petroleum products and lubricants	881.27
Committee D-2	ASTM-IBM	1 137.57
Committee D-2	DCC I Reference fuel account	40 173.74
Committee D-3	DCC II National exchange group	(3 939.50)[a]
Committee D-4	On road and paving materials	998.23
Committee D-5	On coal and coke	1 702.07
Committee D-7	On wood	53.51
Committee D-7	Wood pole research fund	2 097.36
Committee D-7	Wood pole-Program #2	3 608.40
Committee D-8	On bituminous materials for roofing, waterproofing and related building or industrial uses	(11.30)
Committee D-9	On electrical insulating materials	3 516.44
Committee D-10	On shipping containers	12.58
Committee D-11	On rubber and rubber-like materials	812.36
Committee D-11	Rubber special	592.46
Committee D-12	On soaps and other detergents	809.13
Committee D-13	On textile materials	2 278.10
Committee D-14	On adhesives	64.00
Committee D-15	On engine antifreezes	571.01
Committee D-18	On soils for engineering purposes	16.50
Committee D-19	On industrial water	123.69
Committee D-20	On plastics	1 165.05
Committee E-2	On emission spectroscopy	175.12
Committee E-3	On chemical analysis of metals	213.15
Committee E-4	On metallography	40.07
Committee E-9	On fatigue	299.18
Committee E-11	On quality control of materials	1.09
Committee E-14	On mass spectrometry	1 448.06
Committee F-1	On materials for electron tubes and semiconductor devices	219.94
ASA sectional committee on specifications for cast iron pipe and fittings		1 620.51
Chicago district		3 418.31
Cleveland district		211.76
Detroit district		2 430.13
New England district		3 233.82
Northern California district		2 028.62
Ohio Valley district		4.85
Philadelphia district		852.34
Philadelphia district special (annual meeting account)		2 375.16
Pittsburgh district		267.83
St. Louis district		217.96
Southern California district		3 340.73
Southwest district		(17.52)
Washington district		210.02
Western New York-Ontario district		1 793.56
Advisory committee on corrosion		43 243.22
Administrative committee on end-use products		305.37

---

[a] ( ) Indicate red figures.

Joint committee on X-ray diffraction:			
Chemical analysis			74 655.31
Research fellowship			23 902.71
Indexing patterns—IBM calculator			107.71
Joint committee on leather			2.71
Reserve-future West Coast meetings			7 549.85
			246 519.37
Accounted for as follows:			
Cash in bank and on hand		$84 131.05	
Investments (at cost) (market value $38 412.50)			
$20,000 US Treasury 2½s due 8/15/63	$19 925.00		
20,000 US Treasury 2½s due 11/15/61	19 400.00	39 325.00	
Investment fund participation		123 063.32	$246 519.37

DUDLEY MEDAL AND MARBURG LECTURE FUND

Principal Account

Balance, November 27, 1957, Invested	$6 968.07
Receipts:	
Transfer from Income—Stock Dividends	17.26
Transfer from Income—Gain on Investments	434.07
Balance, November 28, 1958, Invested	$7 419.40

Income Account

Balance, November 27, 1957, Cash	505.99	
Receipts:		
Interest, Dividends, and Gain on Investments	750.64	$1 256.63
Disbursements:		
Transfer to Principal Account	451.33	
Award	400.00	
Medals	118.65	
Certificates	92.70	1 062.68
Balance, November 28, 1958, Cash		$193.95

RICHARD L. TEMPLIN AWARD FUND

Principal Account

Balance, November 27, 1957, Invested	$2 229.79
Receipts:	
Transfer from Income—Stock Dividends	5.52
Transfer from Income—Gain on Investments	138.91
Balance, November 28, 1958	$2 374.22

Income Account

Balance, November 27, 1957, Cash	214.74	
Receipts:		
Interest, Dividends, and Gain on Investments	240.21	$454.95
Disbursements:		
Transfer to Principal Account	144.43	
Awards	50.01	
Certificates	3.86	198.30
Balance, November 28, 1958		$256.65

SAM TOUR AWARD FUND

Principal Account

Balance, November 27, 1957, Invested		$1 735.44
Receipts:		
Transfer from Income—Stock Dividends		4.30
Transfer from Income—Gain on Investments		108.10
Balance, November 28, 1958, Invested		$1 847.84

Income Account

Balance, November 27, 1957, Cash	345.72	
Receipts:		
Interest, Dividends, and Gain on Investments	186.94	$532.66
Disbursements:		
Transfer to Principal	112.40	
Awards	35.00	
Certificates	2.57	149.97
Balance, November 28, 1958, Cash		$382.69

F. E. RICHART AWARD

Principal Account

Balance, November 27, 1957, Invested		$1 051.78
Receipts:		
Transfer from Income—Stock Dividends		2.61
Transfer from Income—Gain on Investments		65.51
Balance, November 28, 1958, Invested		1 119.90

Income Account

Balance, November 27, 1957, Cash	45.37	
Receipts:		
Interest, Dividends, and Gain on Investments	113.29	$158.66
Disbursements:		
Transfer to Principal		68.12
Balance, November 28, 1958, Cash		$90.54

ASTM RESEARCH FUND

Principal Account

	Total	*Invested*	*Uninvested Cash*
Balance, November 27, 1957	$84 926.75	$83 171.38	$1 755.37
Transfer from income—stock dividends	193.47	193.47	
Transfer from income—gain on investments	4 864.91	4 864.91	
Transfer from income	3 244.63		3 244.63
Transfer from general fund	3 000.00		3 000.00
Cash invested		5 000.00	(5 000.00)[a]
Balance, November 28, 1958	96 229.76	93 229.76	3 000.00
Investment fund participation	83 155.13		
618 shares General Motors Co (Common) (Note A) (Market value $30,513.75)	7 258.63		
16 shares American Telephone & Telegraph (Note A) (Market value $3,218.00)	2 816.00	93 229.76	

Income Account

Balance, November 27, 1957		10 110.99	
Receipts:			
Interest, dividends and gain on investments		8 412.90	
Transfer from general fund		800.00	19 323.89
Disbursements:			
Transfers to principal		8 303.01	
Grants:			
Corrosion Research Council	300.00		
American Electroplaters' Society	500.00	800.00	9 103.01
Balance, November 28, 1958			$10 220.88

(a) indicate red figures.

Note A—The income from these securities is paid to annuitants.

GENERAL FUNDS: RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD NOVEMBER 27, 1957 TO NOVEMBER 28, 1958

Cash Balance, November 27, 1957, as reported			279 307.37
RECEIPTS			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$284 176.86		
Life Membership	175.00		
Past Dues	1 599.28		
Advance Dues	4 885.45		
Entrance Fees	6 965.00	$297 801.59	
Sales of Publications:			
Book of Standards (Members, Additional Parts)	93 401.31		
1955 Book of Standards	69 885.17		
1958 Book of Standards	20 353.90		
1956 Supplement to Book of Standards	24 307.03		
1957 Supplement to Book of Standards	81 611.54		
Methods of Chemical Analysis of Metals	6 451.78		
Compilation of Standards	119 944.22		
Separate Standards	46 452.44		
Selected Standards for Students	8 144.20		
Proceedings	10 230.77		
Bulletin Subscriptions (Nonmembers)	6 548.69		
Special and Miscellaneous Publications	169 688.79	657 019.84	
Miscellaneous:			
Advertising—BULLETIN	48 112.61		
Advertising—Index	4 399.49		
Interest and Dividends	31 096.93		
Registration and Other Fees—Meetings	16 495.40		
ASTM Exhibit at Annual Meeting	20 244.75		
Staff Service	7 958.08		
Miscellaneous	887.81	129 195.07	
Total Operating Receipts (Budgeted)		1 084 016.50	
Nonoperating Receipts (Not Budgeted):			
Excess Remittances	3 878.54		
Committee C-1 for Technical Assistant	2 846.64		
Sale of X-Ray Diffraction Cards	107 184.93		
From Building Fund on Account of Loan	8 700.00		
From Building Fund on Account of Advance	15 000.00		
From Investment Fund Repayment of Advance	7 000.00		
Loan From Investment Fund	50 000.00		
Annual Meeting Luncheon	2 232.00		
Annual Meeting Buffet Supper	247.25		
St. Louis Luncheon	1 071.00		
Gillett Memorial	490.45		
Mexico City Meeting	406.00		
Gain on Sale of Securities	37 189.57		
Miscellaneous	12.52	236 258.90	
Total Receipts			1 320 275.40
Total Receipts for Fiscal Year 1958 and Cash Balance November 27, 1957			1 599 582.77

Brought Forward			1 599 582.77
DISBURSEMENTS			
Operating Disbursements (Budgeted):			
Publications:			
Year Book	21 436.01		
1955 Book of Standards	16 493.31		
1958 Book of Standards	71 237.93		
1957 Supplement to Book of Standards	12 313.64		
Compilations of Standards	68 310.03		
Separate Standards	17 776.51		
Methods of Chemical Analyses of Metals	14.87		
Proceedings	2 987.88		
Preprints	16 413.85		
ASTM BULLETIN	59 097.92		
Circulars to Members	6 409.97		
Index to Papers	75.00		
Index to Standards	12 304.04		
Special and Miscellaneous and West Coast Publications	154 165.27		
Salaries	406 126.43		
General Office Expenses	104 063.81		
Expenses—Technical and District Committees	14 809.24		
Expenses—Meetings	34 154.30		
Headquarters Occupancy Expense (Includes $10,200 Depreciation on Buildings)	40 143.82		
American Standards Association	2 250.00		
Traveling Expense, Administrative and Special Committees	10 483.76		
Furniture and Fixtures	7 315.77		
Transfer to Principal ASTM Research Fund	3 000.00		
Dues, Contributions, Miscellaneous	3 387.79		
Group Insurance	4 514.55		
Employees' Retirement Fund	33 000.00		
Social Security	6 921.91		
Blue Cross	1 804.32		
Major Medical	2 151.05		
Legal Services	1 250.00		
Total Operating Disbursements (Budgeted)		1 134 412.98	
Nonoperating Disbursements (Not budgeted):			
Refund of Excess Remittances	3 882.44		
Technical Assistant Committee C-1	2 967.10		
Costs and Refunds, X-Ray Diffraction Cards	27 194.70		
Transfer to Joint Committee on X-Ray Diffraction	75 000.00		
Annual Meeting Buffet Supper	308.60		
Annual Meeting Luncheon	3 414.96		
St. Louis Luncheon	1 071.00		
Executive Retirement Reserve	1 200.00		
Gillett Memorial Fund	562.27		
Employee Retirement Reserve	290.64		
Investments	38 668.50		
Advance to Investment Fund	7 000.00		
Meeting, Mexico City	147.40		
1957 Supplement to Book of Standards	47 000.00		
1957 Proceedings	36 000.00		
Deposit on Air Travel	425.00		
Advance to Building Fund	15 000.00		
Collection expense	118.90		
Miscellaneous	322.70	260 574.21	
Total Disbursements			1 394 987.19
Cash Balance, November 28, 1958			$204 595.58

DETAILS OF ASSETS AND LIABILITIES OF BUILDING FUND AS OF NOVEMBER 28, 1958

Assets

Cash in Girard Trust Corn Exchange Bank, Philadelphia, Pa.				$1 202.43
Investment Fund Participation (at cost)				30 401.04
Deposit on Real Estate				15 000.00
Property 1916–1918 Race St., Philadelphia, Pa:				
Cost:				
Land		$30 000.00		
Building	$26 503.35			
Construction (Net of Salvage)	137 295.83			
Additions 1958:				
Partitions	279.00			
Asphalt Tile	395.00			
Light Fixtures	376.00			
	164 849.18			
Accumulated Depreciation (Provided from General Funds)	65 983.05	98 866.13	$128 866.13	
Property 1921–1927 Cherry St. and 1922–1928 Quarry St., Philadelphia, Pa:				
Cost:				
Land		$30 800.00		
Buildings	38 880.10			
Construction	73 030.53			
Additions, 1958:				
Window Guards	348.14			
Venetian Blinds	86.01			
Air Conditioners	2 364.39			
Electric Wiring and Fixtures	275.65			
Partitions	676.00			
Loading Docks	683.00			
	116 343.82			
Accumulated Depreciation	17 816.95	98 526.87	129 326.87	
Property 1919 Cherry St., Philadelphia, Pa:				
Land		7 659.42		
Improvements		890.47	8 549.89	266 742.89
Total Assets				$313 346.36

Liabilities and Funds

Contributions from Members	$188 912.65	
Contributions from ASTM Funds	90 764.57	
Accumulated Net Income	4 344.14	
Loan Payable to ASTM General Funds	29 325.00	$313 346.36

ASTM BUILDING FUND

DETAILS OF ASTM BUILDING FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD NOVEMBER 27, 1957 TO NOVEMBER 28, 1958

Cash Balance, November 27, 1957		$11 377.71
Receipts		
Contributions from Members	$4 959.60	
Interest and Dividends on Investments	1 297.13	
Profit on Sale of Investments	1 778.58	
From ASTM General Fund:		
On Account of Depreciation on Building, Elevator, and Air Conditioning Installation	10 200.00	
On Account of Amortization of Note	4 350.00	
		22 585.31
		33 963.02
Disbursements		
Expenses, Cherry Street Property:		
Real Estate Taxes, 1958	2 711.15	
Construction	4 433.19	
Expense—Moving Department	306.94	
Expenses, Race Street Property:		
Construction	1 050.00	
Reduction of Loan from ASTM General Fund	4 350.00	
Transferred to Investment Fund	4 849.31	
Deposit on Real Estate	15 000.00	32 760.59
Cash Balance, November 28, 1958		1202.43

ASTM INVESTMENT FUND

AS OF NOVEMBER 17, 1958

Cash	$33 036.36
Investments (at cost) (Market value $1 011 810.85)	802 020.32
Loan Receivable General Fund	50 000.00
Total Assets	$885 056.68
Participation by Funds	885 056.68

PARTICIPATION BY FUNDS

	Participation November 15, 1957	*Additional Funds Invested*	*Income Reinvested*	*Net Profit, Sale of Securities*	*Total Participation November 17, 1958 (At Cost)*
General fund	$597 007.33		$1 478.93	$37 189.57	$635 675.83
Committee funds	108 077.31	$7 500.00	286.31	7 199.70	123 063.32
Research fund	73 096.75	5 000.00	193.47	4 864.91	83 155.13
Building fund	25 551.73	3 000.00	70.73	1 778.58	30 401.04
Medal and Lecture fund	6 968.07		17.26	434.07	7 419.40
Templin Award fund	2 229.79		5.52	138.91	2 374.22
Sam Tour fund	1 735.44		4.30	108.10	1 847.84
Richart fund	1 051.78		2.61	65.51	1 119.90
	$815 718.20	$15 500.00	$2 059.13	$51 779.35	$885 056.68

ASTM INVESTMENT FUND

DETAILS OF INVESTMENT FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD NOVEMBER 16, 1957 TO NOVEMBER 17, 1958

Cash Balance, November 15, 1957			$28 182.35
Receipts			
Interest and Dividends on Investment	$39 824.13		
Interest on Savings Accounts	226.00		
Interest on Certificates of Deposit	200.00		
Total Interest and Dividends		$40 250.13	
Proceeds from Sales of Investments	168 398.07		
Additional Funds Invested by Participants	15 500.00		
Reinvestment of Stock Dividends by Participants	2 059.13		
Loan from General Funds	7 000.00	192 957.20	
Total Receipts			233 207.33
			261 389.68
Disbursements			
Purchase of Securities		131 090.49	
Investment Counsel		2 500.00	
Repayment of Loan from General Funds		7 000.00	
Loan to General Fund		50 000.00	
Distribution of Interest and Dividends to Participants:			
General Fund	27 122.47		
Committee Funds	5 250.75		
Research Fund	3 547.99		
Building Fund	1 297.13		
Medal and Lecture Fund	316.57		
Templin Award Fund	101.30		
Sam Tour Fund	78.84		
Richart Fund	47.78	37 762.83	
Total Disbursements			228 353.32
Cash Balance, November 17, 1958			33 036.36
Balance Composed of:			
Demand Account		30 157.64	
Savings Account		2 878.72	$33 036.36

LIST OF SECURITIES—INVESTMENT FUND

As of November 17, 1958

Investments

Number of Shares or Par Value	*Bonds*	*Cost or Book Value*	*Market Value*[a]
10 000	Commercial Credit Co. 4½ per cent 1977	9 806.00	9 400.00
15 000	Consolidated Edison Co. of N. Y., Inc. 1st and Ref. 3⅝ per cent Series L 5/1/86	15 206.55	13 500.00
15 000	Consumers Power Co. 4¾% 10/1/87	15 187.50	15 675.00
15 000	General Motors Corp. 3¼ per cent Deb. 1/1/79	14 925.00	13 800.00
10 000	General Telephone Corp. 4½ per cent Conv. Deb. 6/1/77	10 681.23	12 475.00
5 000	Pacific Gas & Electric Co. 1st and Ref. 3⅛s Series W 12/1/84	5 150.78	4 168.75
10 000	Radio Corp. of America 3½ per cent Conv. Deb. 12/1/80	11 075.00	10 025.00
20 000	Scott Paper Co. 3 per cent Conv. Deb. 3/1/71	20 377.80	21 650.00
10 000	Sinclair Oil Corp. 4⅜ per cent 1986	11 356.68	11 675.00
15 000	Socony Mobil Co. 2½ per cent Deb. 6/1/76	13 995.95	12 187.50
15 000	Southern Natural Gas Co. 1st Pipe Line S F 3⅛s 2/1/74	14 734.81	12 750.00
15 000	Southwestern Bell Telephone Co. 4¾% debs 10/1/92	15 450.00	15 825.00
15 000	Superior Oil Co. 3¾ per cent Deb. 7/1/81	15 000.00	13 875.00
10 000	Tennessee Gas Transmission Co. 1st Mtg Pipe Line 4¾s 11/1/76	10 065.00	9 800.00
10 000	U. S. Federal Land Banks 4% 9/20/61	10 027.00	10 062.50
10 000	U. S. Federal Land Banks 3⅞ per cent 9/15/72	9 806.25	9 750.00
15 000	U. S. Twelve Federal Land Banks 4⅛s 2/15/72	15 000.00	14 962.00
15 000	Federal National Mortgage Association 4⅛% 11/12/63	15 002.00	14 990.63
15 000	U. S. Treasury 2½s 11/15/61	15 000.00	14 550.00
15 000	U. S. Treasury 2½s 6/15/67–72	14 694.08	13 256.25
15 000	U. S. Treasury 2½s 8/15/63	14 943.75	14 259.38
15 000	U. S. Treasury 2¾s 9/15/61	14 934.38	14 737.50
20 000	U. S. Treasury 4s 8/1/61	20 056.25	20 325.00
30 000	U. S. Savings Bonds Series G 2½s 6/1/61	30 000.00	29 280.00
5 000	U. S. Savings Bonds Series G 2½s 1/1/62	5 000.00	4 850.00
5 000	U. S. Savings Bonds Series G 2½s 2/1/62	5 000.00	4 850.00
	Total Bonds	352 476.01	342 680.01
	Preferred Stocks		
Shares			
30	Endicott Johnson Corp. 4 per cent Pfd	2 937.50	2 460.00
30	General Motors Corp. $5 Series Pfd	3 755.80	3 270.00
30	Long Island Lighting Co. 4.35 per cent Series E Pfd	3 015.00	2 565.00
30	P. Lorillard Co. 7 per cent Pfd	4 380.00	4 140.00
30	Mississippi Power & Light Co. 4.36 per cent Cum Pfd	3 075.00	2 550.00
100	Philadelphia Electric Co. 4.40 per cent Cum Pfd	10 670.00	9 750.00
47	Standard Slag Co. 2nd Pfd	2 115.00	2 115.00
30	Westinghouse Electric Corp. 3.80 per cent Cum Pfd Series B	3 067.74	2 595.00
	Total Preferred Stocks	33 016.04	29 445.00

---

[a] All market values of securities are as of November 15, 1958.

Common Stocks

Shares			
150	Abbott Laboratories	$8 884.37	$10 500.00
202	Allegheny Ludlum Steel Corp.	8 111.87	9 822.25
100	Aluminum Co. of America	2 061.45	8 700.00
200	Aluminum, Ltd.	6 922.26	6 400.00
100	American Can Co.	3 884.25	5 200.00
400	American Metal Climax, Inc.	7 271.86	11 250.00
100	American Radiator & Standard Sanitary Co.	1 564.32	1 375.00
175	American Stores Co.	7 689.41	16 318.75
100	American Telephone and Telegraph Co.	17 465.36	20 112.50
160	Bethelehem Steel Corp.	2 095.99	8 160.00
136	Chase Manhattan Bank	3 930.06	7 854.00
200	Cleveland Electric Illuminating Co.	8 271.00	9 100.00
300	Consolidation Coal Co.	12 071.32	11 850.00
100	Crown Zellerbach Corp.	2 914.79	5 712.50
75	Delaware Power and Light Co.	2 454.11	4 406.25
150	Denver and Rio Grande Western Railroad Co.	7 701.73	8 137.50
171	Dow Chemical Co.	4 034.38	13 038.75
50	E. I. duPont de Nemours & Co.	6 544.18	10 325.00
50	Florida Power & Light Co.	1 969.44	4 200.00
125	General Electric Co.	4 311.15	8 750.00
150	General Mills, Inc.	10 630.89	12 037.50
225	General Telephone Corp.	3 312.82	12 937.50
150	Great Northern Paper Co.	12 688.34	7 500.00
142	Gulf Oil Corp.	3 505.03	17 679.00
238	Houston Lighting & Power Co., Texas	8 061.31	17 671.50
200	International Harvester Co.	6 406.67	8 250.00
200	Johns-Manville Corp.	8 063.78	10 300.00
107	Jones & Laughlin Steel Corp.	2 670.72	6 139.13
500	Lone Star Cement Corp.	4 941.44	17 437.50
100	Merck & Co., Inc.	3 887.50	7 512.50
50	National Lead Co.	5 059.25	5 525.00
103	Niagara Mohawk Power Corp.	3 479.57	4 004.13
200	Ohio Oil Corp.	2 623.35	8 025.00
60	Pacific Gas & Electric Co.	2 301.42	3 720.00
321	Philadelphia Electric Co.	8 599.20	14 605.50
200	Republic Steel Corp.	8 523.26	13 975.00
100	Sherwin Williams Co.	10 515.69	19 500.00
138	Socony Mobil Oil Co.	3 103.03	6 848.25
150	Southern Railway Co.	5 998.81	8 025.00
153	Square D Co.	2 362.37	4 360.50
200	Standard Oil of Indiana	11 969.64	9 675.00
285	Standard Oil of New Jersey	8 971.97	17 064.38
282	Texas Co.	14 165.58	24 146.25
60	Union Carbide Corp.	4 235.24	7 192.50
240	Westinghouse Electric Co.	15 644.26	16 290.00
220	Wisconsin Electric Power Co.	7 368.63	7 700.00
	Total Common Stocks	299 243.07	469 333.64

Mutual Funds

Shares			
4 598.718	Commonwealth Investment Co.	33 852.16	44 607.56
5 376	Eaton Howard Balanced Fund	83 433.04	125 744.64
	Total Mutual Funds	117 285.20	170 352.20

Summary

Bonds	352 476.01	342 680.01
Preferred Stocks	33 016.04	29 445.00
Common Stocks	299 243.07	469 333.64
Mutual Funds	117 285.20	170.352.20
	$802 020.32	$1 011 810.85

NOTE: Income includes proceeds from sales of rights.

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

The Administrative Committee on Standards again has had a busy year as indicated below. In fact, the number of items requiring attention seems to increase from year to year. Partly in view of this, additions have been made to the personnel of the committee, the present membership being as follows:

J. H. Foote (Chairman), Commonwealth Associates, Inc.
G. H. Harnden, General Electric Co.
L. J. Jacobi, The Detroit Edison Co.
W. A. Kirklin, Hercules Powder Co.
J. L. Menson, Combustion Engineering, Inc.
M. A. Pinney, The Pennsylvania Railroad Co.
J. B. Rather, Jr., Socony Mobil Oil Co., Inc.
L. S. Reid[a] (Retired), Metropolitan Life Insurance Co.
F. W. Reinhart, National Bureau of Standards
S. A. Rosecrans, Westinghouse Electric Corp.
I. V. Williams, Bell Telephone Laboratories, Inc.

The Administrative Committee is interested in all aspects of the Society's standardization work. The several functions are discussed under the subdivision headings of this report.

Actions by Standards Committee on Committee Recommendations:

The review of recommendations from technical committees is, of course, one of the primary functions of the Standards Committee, since in its establishment it was intended to provide a means of having recommendations acted upon in the interval between annual meetings. Many of the annual reports refer to recommendations that have been submitted to the Standards Committee during the year. The actions taken this year by the Standards Committee are summarized below:

New Tentatives	52
Revised Tentatives	67
Tentative Revisions of Standards	5
Standards Revised and Reverted to Tentative	12
Tentatives Withdrawn	1
Emergency Alternate Provisions Withdrawn	2

A number of these recommendations were acted upon at meetings held on September 9, 1958, and March 25, 1959; the remainder were handled by correspondence.

Actions by the Society at 1958 Annual Meeting:

As a matter of record, the actions taken by the Society at the 1958 Annual Meeting with respect to standards and tentatives are tabulated below:

New Standards and Existing Tentatives Adopted as Standard	137
Standards Revised	166
New Tentatives	112
Standards Revised and Reverted to Tentative	22
Tentative Revisions of Standards	16
Tentatives Revised	158
Standards and Tentatives Withdrawn	21

Promotion of Standardization Work:

The first assignment of the Standards Committee as set forth in its scope is "to promote the standardization work of the Society." This can take several forms, including the extension of standardization activities as set forth below.

---

[a] Subsequent to the presentation of this report, advice was received of the death, on July 15, 1959, of Mr. Reid.—Ed.

The Standards Committee, however, has reviewed its assignment and is recommending a number of ways in which it believes the Society's standardization work can be promoted in one way or another. These recommendations have been referred to the Board for its consideration and development. Included in the proposals is the sponsoring of discussions on various aspects of the Society's standardization work, and various possible means of promoting the use of ASTM standards.

Recommendations on Form of ASTM Standards:

The Standards Committee has repeatedly called attention to the desirability of having greater attention given on the part of the technical committees to the form of specifications and test methods. A recommended form of specifications was made available to the committees several years ago and this is now under revision in the light of various suggestions received.

A companion Recommended Form of Methods of Test is now in preparation in Committee E-1 on Methods of Testing. It is expected that this will include some discussion of the types of method appropriate for publication as ASTM standards. The Administrative Committee on Standards has given a great deal of consideration to this subject in the light of the various methods that are submitted to it. It is making the following recommendation to the Board: "It is the consensus of the Standards Committee that there need be no limitations put on the type of test published by the Society but that the application of the methods should be carefully explained in the scope."

More Adequate Report From Technical Committees:

The Standards Committee has had occasion to comment at various times in connection with recommendations submitted to it on the very incomplete return on letter ballots. The Standards Committee as a guide in its own operations has required a 50 per cent return on letter ballots in the committee before it will consider any recommendation. This would appear to be a very low return to serve as a basis for approval of a proposed standard, and it believes consideration might well be given to including a requirement in the general Regulations Governing Technical Committees that a more adequate return on letter ballots is required. It is understood this is receiving consideration in the Long-Range Planning Committee.

EXPANSION OF STANDARDIZATION ACTIVITIES

An important function of the Standards Committee is to explore with the Board of Directors proposals dealing with new projects. Comments with respect to a number of these are set forth below.

Industrial Chemicals.—Reference was made in the report of the committee a year ago of considerations being given to the establishment of a new Committee E-15 on Industrial Chemicals. This committee has now been organized and one of its principal functions will be the development of methods of testing, particularly such methods as have rather general applicability. It will also function in a reviewing and coordinating capacity, and possibly in recommending new standardization activities in the industrial chemical field.

Industrial Pitches.—A need developed for specifications for bituminous fiber pipe since specifications for this material have been developed by the industry and provision should be made for these specifications in ASTM. Upon bringing this subject to the attention of Committee D-8 on Bituminous Waterproofing and Roofing Materials, the committee took jurisdiction over the bituminous fiber pipe and extended it to include industrial pitches in general.

Cleaning Solvents.—Mention has been made before of considerations being given to undertaking work in the field of cleaning solvents, both from the point of view of cleaners for electric motors and also other types of cleaning such as that utilizing vapor degreasers. On the recommendation of the committee, a new committee was authorized and the organization meeting of Committee D-26 on Halogenated Organic Solvents was held during the 1958 Annual Meeting of the Society in Boston.

Cermets.—After very complete exploration, a study committee is recommending to the Board that no new technical committee on cermets be established at this time but that a coordinating committee be set up made up of representatives of the several ASTM committees interested, with the thought that this coordinating committee would keep the Board currently informed concerning any further needs in this field.

Gaskets and Packing Materials.—A study committee has been functioning during the past year, examining into the needs for broader coverage in the field of gaskets and packing materials than that currently provided in Committee D-11 on Rubber and Rubber-like Materials. An extensive questionnaire has been conducted but as yet no specific recommendations have been made in the light of this questionnaire as to how this work should be organized.

Ore Sampling and Analysis.—As the result of further inquiry, there would seem to be definite interest in having work organized under the Society on sampling and analysis of ores. The present inquiry was prompted in view of the interest in manganese ores, but the inquiry has included a much broader coverage including fluxes and similar materials. A conference is being held on June 2, 1959, to give consideration to the specific needs and whether a new technical committee should be established or whether the work might be assigned to one or more of the existing ASTM committees as, for example, Committee E-3 on Chemical Analysis of Metals.

Graphite.—The nuclear uses of graphite have pointed up the need for specifications in this field. Other structural uses of graphite are assuming increased importance. Consideration is accordingly being given to setting up a working group on graphite, having in mind as its first objective standards in the nuclear field. This may subsequently be extended to other uses of graphite. The present considerations in the Society are confined to the use of graphite in lubricants.

Caulking Compounds and Sealants.—Committee D-1 on Paint, Varnish, Lacquer and Related Products has had a subcommittee at work on putty, glazing, and caulking compounds. These for the most part have been of the oil and pigment types, and the work so far has been concentrated in the development of methods of testing. More recently there has been an interest in the industry for specifications covering the elastomeric type of caulking compounds. Consideration is now being given as to how the Society might best organize the work so as to include this type of compound as well, particularly since there is urgent demand for the standards in this field.

Pigments.—While work on testing of pigments is of long standing in the Society under Committee D-1 on Paint, Varnish, Lacquer and Related Products, some interest has been expressed in the extension of this to include pigments as used in other materials such as paper, plastics, and rubber.

RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Two methods under the procedure of the American Standards Assn. are used by ASTM in submitting recommendations to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing ASTM standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the ASTM is granted proprietary sponsorship insofar as any future revisions are concerned, and such revisions are brought to the attention of the ASA currently for approval under this proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations, usually with the ASTM as sponsor or co-sponsor of the sectional committee.

Standards Submitted Under the Existing Standards Procedure:

Eighteen standards relating to plastics were submitted to the American Standards Association for approval as American Standard under the existing standards procedure.

Revised Standards Submitted Under the Proprietary Procedure:

In view of the Society having been designated as proprietary sponsor for the revisions of ASTM standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Assn. during the year revisions of 12 standards relating to steel, 1 relating to corrosion of iron and steel, 4 relating to non-ferrous metals and alloys, 3 relating to electrodeposited metallic coatings, 5 relating to copper, 2 to gypsum, 2 to refractories, 2 to manufactured masonry units, 2 to linseed oil, 1 to wood, 1 to rubber, 1 to engine antifreezes, 1 to turpentine, 1 to detergents, 1 to thermometers, and 1 to fire tests.

Standards Submitted Under the Sectional Committee Procedure:

During the year, the following sectional committees, for which the Society is sponsor or co-sponsor, took action to present recommendations to the ASA as follows:

Sectional Committee A1 on Hydraulic Cements.—This committee has recommended the approval as American Standard of 2 ASTM standards, the approval of revisions of 2 American Standards, and the reaffirmation of 3 American Standards.

Sectional Committee A88 on Magnesium Oxychloride Cement Flooring.—This committee has recommended the approval of revisions of 1 American Standard.

Sectional Committee B36 on Wrought-Iron and Wrought-Steel Pipe and Tubing.—This committee has recommended the approval of revisions of 15 American Standards.

Sectional Committee C7 on Bare Electrical Conductors.—This committee has recommended the approval of revisions of 11 American Standards.

Sectional Committee C59 on Electrical Insulating Materials.—This committee has recommended the approval as American Standard of 9 ASTM standards, and the approval of revisions of 1 American Standard.

Sectional Committee Z11 on Petroleum Products and Lubricants.—This committee has recommended the approval as American Standard of 2 ASTM standards, the approval of revisions of 11

American Standards, and the reaffirmation of 1 American Standard.

Sectional Committee Z23 on Sieves for Testing Purposes.—This committee has recommended the approval of revisions of 1 American Standard.

INTERNATIONAL RELATIONS

The international discussions, particularly under the International Organization for Standardization (ISO) and the International Electrotechnical Commission (IEC), are assuming ever greater significance. A number of the European countries apparently wish to have the standardization discussions handled internationally and much of the work is supported here rather than in the development of individual national standards. In certain areas these international discussions are having a definite impact on the standardization in this country, and a number of ASTM committees are following these international discussions quite closely with the thought that the ASTM standards and the international recommendations should be in harmony. A list of the various ISO and IEC projects in which ASTM is interested, which continues to grow year by year, is given below.

ISO Projects:

Aromatic Hydrocarbons.—ASTM Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials is handling participation by the United States in the work of ISO/TC 78 on Aromatic Hydrocarbons for which Great Britain holds the secretariat.

Asbestos-Cement Products.—ASTM Committee C-17 on Asbestos-Cement Products has indicated that it favors American participation in the work of ISO/TC 77 on Asbestos-Cement Products and that it is prepared to be responsible for the participation by the United States in this work.

Cast Iron.—Great Britain holds the secretariat for ISO/TC 25 on Cast Iron. The Advisory Committee of ASTM Committee A-3 on Cast Iron has been designated to handle American participation.

Chemistry.—Italy holds the secretariat for ISO/TC 47 on Chemistry. Several ASTM Committees have indicated an interest in this work, and draft proposals which have been received have been passed on to these committees.

Copper and Copper Alloys.—The United States holds the secretariat for ISO/TC 26 on Copper and Copper Alloys. The Advisory Committee of ASTM Committee B-5 on Copper and Copper Alloys has been designated to handle the work of the secretariat.

Fibre Building Board.—Consideration is being given by ASTM Committee D-7 on wood to the desirability of participation by the United States in the work of ISO/TC 89 on Fibre Building Board.

Fire Tests for Building Materials and Structures.—ASTM Committee E-5 on Fire Tests of Materials and Construction has recommended against United States participation in the work of ISO/TC 92 on Fire Tests for Building Materials and Structures on the basis that such participation could not be justified in view of the expense involved.

Hydraulic Binders.—Sectional Committee A1 on Hydraulic Cements, for which ASTM is sponsor, has recommended participation by the United States in the work of ISO/TC 74 on Hydraulic Binders for which Belgium holds the secretariat. The sectional committee further recommended that ASTM Committee C-1 on Cement handle the United States participation in this work.

Iron and Steel.—The United States has the status of an observer in the work of ISO/TC 17 on Iron and Steel for which Great Britain holds the secretariat. A number of draft proposals have been received which have been brought to the attention of ASTM Committee A-1 on Steel, while others dealing with methods of testing have been submitted to the appropriate subcommittees of ASTM Committee E-1 on Methods of Testing.

Light Metals and Alloys.—The Advisory Committee of ASTM Committee B-7 on Light Metals and Alloys, Cast and Wrought, has been designated to handle American participation in the work of ISO/TC 79 on Light Metals and Alloys. France holds the secretariat for this ISO Committee.

Mica.—Subcommittee IX on Mica of ASTM Committee D-9 on Electrical Insulating Materials has been set up to handle the contacts by the United States with ISO/TC 56 on Mica for which India holds the secretariat.

Paper.—ASTM Committee D-6 on Paper and Paper Products has recommended participation by the United States in the work of ISO/TC 6 on Paper for which France holds the secretariat.

Petroleum Products.—The United States holds the secretariat for ISO/TC 28 on Petroleum Products, the technical work of the secretariat being under the purview of Sectional Committee Z11 on Petroleum Products and Lubricants, for which ASTM is sponsor.

Plastics.—America holds the secretariat for ISO/TC 61 on Plastics. ASTM Committee D-20 on Plastics is handling the work of the secretariat and has organized for the purpose a special committee consisting of representatives of the various groups interested.

Raw Materials for Paints, Varnishes, and Similar Products.—ASTM Committee D-1 on Paint, Varnish, Lacquer and Related Products is handling United States contacts with ISO/TC 35 on Raw Materials for Paints, Varnishes, and Similar Products, for which the Netherlands holds the secretariat.

Refractories.—ASTM Committee C-8 on Refractories is handling United States contacts with ISO/TC 33 on Refractories for which Great Britain holds the secretariat.

Rubber.—The secretariat for ISO/TC 45 on Rubber is held by Great Britain. The American group to handle participation in this ISO committee has been set up under ASTM Committee D-11 on Rubber and Rubber-like Materials.

Shellac.—Subcommittee XIII on Shellac of ASTM Committee D-1 on Paint, Varnish, Lacquer and Related Products has been set up as the American committee in charge of contacts with ISO/TC 50 on Lac for which India holds the secretariat.

Sieves.—Sectional Committee Z23, for which the Society is co-sponsor with the National Bureau of Standards, handles the contacts for the United States in the work of ISO/TC 24 on Sieves.

Solid Mineral Fuels.—ASTM Committee D-5 on Coal and Coke is handling the contacts of the United States with ISO/TC 27 on Solid Mineral Fuels for which Great Britain holds the secretariat.

Surface Active Agents.—ASTM Committee D-12 on Soaps and Other Detergents has recommended participation by the United States in the work of ISO/TC Committee on Surface Active Agents. This has, however, not yet been cleared in the American Standards Association, particularly as to organization of the United States group to handle this participation.

Statistical Treatment of Series of Observations.—The Netherlands holds the secretariat for ISO/TC 69 on Statistical Treatment of Series of Observations. The United States is participating in this work through a special group on which ASTM is represented.

Textiles.—Great Britain holds the general secretariat for ISO/TC 38 on

Textiles but the United States holds the secretariat for several subcommittees. Participation by the United States in this work is being handled by a special committee organized under the sponsorship of ASTM and the American Association of Textile Chemists and Colorists with representation from the various groups interested. This special committee has been given the designation of L23.

Viscosity.—The United States holds the secretariat for ISO/TC 66 on Viscosity. The Subcommittee on Rheological Properties of ASTM Committee E-1 on Methods of Testing has been set up to handle the work of the secretariat.

Zinc.—ASTM Committee B-2 on Non-Ferrous Metals and Alloys favors participation by the United States in the work of ISO/TC 18 on Zinc for which Belgium holds the secretariat.

IEC Committees:

Insulating Oils.—F. M. Clark, chairman of ASTM Committee D-27 on Electrical Insulating Liquids and Gases, is the technical advisor to the U. S. National Committee of the International Electrotechnical Commission, in connection with the work of IEC/TC 10 on Insulating Oils.

Insulating Materials.—Arnold H. Scott of the National Bureau of Standards is technical advisor to the U. S. National Committee of the IEC for IEC/TC 15 on Insulating Materials. He looks to ASTM Committee D-9 on Electrical Insulating Materials for assistance in processing recommendations from this IEC committee.

Magnetic Steel.—The technical advisor to the U. S. National Committee of the IEC for the work of IEC/TC 14A on Magnetic Steel is A. C. Beiler, chairman of ASTM Committee A-6 on Magnetic Properties.

Aluminum.—E. J. Holcomb of the Aluminum Company of America is technical advisor to the U. S. National Committee for IEC/TC 7 on Aluminum. Mr. Holcomb looks to Sectional Committee C7, for which ASTM is sponsor, for assistance in processing recommendations from this IEC committee.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
Chairman.

R. E. HESS,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

Mention has been made in recent reports of the Administrative Committee on Papers and Publications of the increase in work facing the committee. In view of this, some enlargement has been made in the personnel so that the committee now comprises ten appointees. The present membership is as follows:

Robert Burns, National Research Council
C. A. Carpenter, Bureau of Public Roads
Thomas Hazen, Union Carbide Plastics Co.
F. M. Howell, Aluminum Company of America
W. T. Lankford, U. S. Steel Corp.
William Lerch, Portland Cement Assn.
E. C. Shuman, Owens-Corning Fiberglas Corp.
A. C. Smith, Atlantic Refining Co.
C. R. Sutton, International Nickel Co.
R. J. Wirshing, General Motors Corp.

Messrs. L. L. Wyman, National Bureau of Standards; L. W. Wood, Forest Products Laboratory; and T. J. Dolan, University of Illinois, have retired after serving two three-year terms.

The additional burden placed upon the members of the committee is due to two things: the increasing number of papers which the Society is asked to publish and the ever-increasing emphasis on economy in publication. The Papers Committee has been ever alert to the desirability of having concise presentation and recommending condensation wherever appropriate, but apart from this all publication activities are being carefully scrutinized at the request of the Board of Directors. As a result, a considerable amount of material is being withheld from preprints for the current Annual Meeting—for example, material that is published as information in committee reports that is not essential to the consideration of these reports at the Annual Meeting. Consideration is also being given to withholding any material from the *Proceedings* that appears in other publications and can be referred to in this way.

The committee is also recommending that economy be effected in the Year Book through placing the Geographic Distribution of Members as well as the listing of subcommittee personnel on a biennial basis. In other words, the complete personnel of subcommittees will appear in alternate years; in the intervening year the Year Book will contain, in addition to the personnel of the main committees, only a listing of the subcommittees and their officers.

What has contributed at the present time to the amount of items to be considered by the Papers Committee is the organization of the Third Pacific Area National Meeting to be held in San Francisco in October. Largely as a result of proposals from the technical committees, the number of symposiums and special sessions scheduled number 53. This will necessitate as many as five and sometimes six concurrent sessions. The number of papers is well over 200. In connection with all of the technical committee-sponsored sessions, a special program or symposium committee functions on which the West Coast committee is represented as well as the Committee on Papers and Publications. In some instances this representation is handled by former members of the Papers Committee.

ASTM Bulletin:

The question of whether or not a reader survey should be conducted has been discussed from time to time. The primary objective of the BULLETIN is to serve as a news medium for the Society, including information about publications, committees, technical activities, and meetings. A secondary objective is to provide technical papers of rather broad and general interests, book reviews, and similar articles. The third objective and one that is rather overriding is to serve as a place for cross fertilization of ideas through both the news stories and the technical papers. It was the consensus in the staff that we were meeting these objectives fairly well but that perhaps we were not doing all that is implied; a survey might help us to do a better job if significant information is uncovered. It was decided that a limited survey would be worth while; the Papers Committee recommended that this be done on a step by step basis, reviewing the information received with some statisticians and then planning any further survey.

Two meetings of the Administrative Committee were held during the year: one on November 12, 1958 and the other on February 12, 1959.

The extent of the Society's publications is indicated in the record of publications issued during the year.

1959 Annual Meeting:

A full program was planned for the 1959 Annual Meeting consisting of some 36 sessions; of these 13 were devoted to special symposiums as follows:

Symposium on Education in Materials,
Symposium on Methods of Test for Design of Bituminous Paving Mixtures,
Symposium on Time Rates of Loading in Soil Testing,
Symposium on Atterberg Limits,
Symposium on Practical and Statistical Significance of Tests and Properties of Bituminous Binders,
Symposium on Spectroscopic Excitation Sources,
Symposium on Microscopy,
Symposium on Identification of Water-Formed Deposits, and
Symposium on Visual Aids for Standardizing and Communicating Product Appearance.

RECORD OF PUBLICATIONS ISSUED
(April 15, 1958 to April 15, 1959)

Regular Publications:

1958 Proceedings, 1430 pp., 7500 copies.
1958 Gillett Lecture, 22 pp., 3000 copies.
1958 Book of ASTM Standards:
    Part 1, 1524 pp., 23,000 copies.
    Part 2, 1388 pp., 21,000 copies.
    Part 3, 994 pp., 20,000 copies.
    Part 4, 1460 pp., 18,500 copies.
    Part 5, 1144 pp., 15,500 copies.
    Part 6, 1040 pp., 12,000 copies.
    Part 7, 1520 pp., 13,000 copies.
    Part 8, 1632 pp., 13,000 copies.
    Part 9, 2050 pp., 16,500 copies.
    Part 10, 1526 pp., 13,000 copies.
1958 Year Book, 784 pp., 8300 copies.
1958 Index to Standards, 240 pp., 24,000 copies.
ASTM BULLETIN, 8 issues (May 1958 to April 1959), total number of pages 844, average number of copies 16,125 (278 pages of technical papers).

A statement of the volume of the principal publications for the past four years is given below:

	1955–1956, pages	1956–1957, pages	1957–1958, pages	1958–1959, pages
Proceedings	1128	1528	1440	1430
Marburg Lecture	54	51	40	....
Gillett Lecture	38	32	36	22
Book of ASTM Standards	11 752	....	....	14 390
Supplements to Book of Standards	....	2335	2644	....
ASTM Methods of Chemical Analysis of Metals	....	640	....	....
Index to ASTM Standards	216	240	251	240
Year Book	620	640	756	784
* Special Technical Publications	3018	2939	3557	3693
* Other Special Publications	....	108	88	40
ASTM BULLETIN	808	836	840	844
Totals	17 634	9349	9652	21 443

*** Not included under this listing are a number of publications which are partly of the nature of compilations of standards such as:**

Building Code Compilation,
Manual of ASTM Standards on Refractory Materials,
Procedures for Testing Soils,
Manual on Industrial Water (STP 148),
Selected ASTM Engineering Materials Standards for Use in College Curricula,
Manual on Measurement and Sampling of Petroleum and Petroleum Products, and
Knock Test Manuals.

Special Compilations of Standards Published from April 1958 to April 15, 1959:

Specifications for Steel Piping Materials (A-1), 480 pp., 3000 copies.
ASTM Standards on Light Metals and Alloys (B-7), 347 pp., 1000 copies.
ASTM Standards on Electrodeposited Metallic Coatings (B-8), 124 pp., 4500 copies.
ASTM Standards on Cement (C-1), 280 pp., 3000 copies.
ASTM Standards on Mineral Aggregates, Concrete, and Nonbituminous Highway Materials (C-9, D-4), 387 pp., 2750 copies.
ASTM Standards on Paint, Varnish, Lacquer and Related Products (D-1), 1052 pp., 2000 copies.
ASTM Standards on Petroleum Products and Lubricants (D-2), 1244 pp., 7500 copies.
ASTM Standards on Gaseous Fuels (D-3), 204 pp., 1250 copies.
ASTM Standards on Bituminous Materials for Highway Constructions, Waterproofing and Roofing (D-4, D-8), 457 pp., 2000 copies.
ASTM Standards on Rubber and Rubber-Like Materials (D-11), 908 pp., 3000 copies.
ASTM Standards on Soaps and Other Detergents (D-12), 255 pp., 1200 copies.
ASTM Standards on Textile Materials (D-13), 880 pp., 2300 copies.
ASTM Procedures for Testing Soils (D-18), 552 pp., 6000 copies.
ASTM Standards on Plastics (D-20), 1108 pp., 4000 copies.
Building Code Compilation, 1065 pp., 3000 copies.
Manual on Measurement and Sampling of Petroleum and Petroleum Products, 166 pp., 4000 copies.
ASTM Manual for Rating Aviation Fuels by Supercharge and Aviation Methods, 333 pp., 1500 copies.
Selected ASTM Engineering Materials Standards for Use in College Curricula, 380 pp., 15,000 copies.

Special Compilations in Prospect:

Specifications for Steel Piping Materials (A-1), 524 pp., 3000 copies.
ASTM Standards on Wood (D-7), 452 pp., 1600 copies.

Special Publications Issued from April 15, 1958 to April 15, 1959:

Codes and Instructions for Wyandotte ASTM Punched Cards, 40 pp., 1000 copies.
Bibliography and Abstracts on Electrical Contacts (STP 56-L), 53 pp., 1500 copies.
Bibliography of Fatigue References (STP 9-I), 64 pp., 500 copies.
Tentative Guide for Fatigue and Statistical Analysis of Fatigue Data (STP 91-A), 90 pp., 1500 copies.
Symposium on Radiation Effects on Materials—Volume II (STP 220), 153 pp., 2500 copies.
Symposium on Spectrochemical Analysis for Trace Elements (STP 221), 85 pp., 2000 copies.
Symposium on Determination of Gases in Metals (STP 222), 66 pp., 2000 copies.
Symposium on Nondestructive Tests in the Nuclear Energy Field (STP 223), 403 pp., 3000 copies.
Symposium on Composition of Petroleum Oils (STP 224), 292 pp., 2500 copies.
Knocking Characteristics of Pure Hydrocarbons (STP 225), 100 pp., 3000 copies.
The Elevated Temperature Properties of Weld-Deposited Metal and Weldments (STP 226), 237 pp., 3000 copies.
Elevated Temperature Properties of Chromium Steels (STP 228), 117 pp., 3000 copies.
Symposium on Effect of Ozone on Rubber (STP 229), 136 pp., 2400 copies.
Symposium on Elevated Temperature Strain Gages (STP 230), 167 pp., 3000 copies.
Symposium on Brittle Failure of Rotor Forgings (STP 231), 49 pp., 2000 copies.
Conference Papers on Soils for Engineering Purposes (STP 232), 507 pp., 1200 copies.
Symposium on Radiation Effects on Materials—Volume III (STP 233), 168 pp., 2500 copies.
Symposium on Radioactivity in Industrial Water and Industrial Waste Water (STP 235), 75 pp., 2000 copies.
Symposium on Some Approaches to Durability in Structures (STP 236), 71 pp., 2000 copies.
Symposium on Basic Mechanisms of Fatigue (STP 237), 128 pp., 2500 copies.
Symposium on Solvent Extraction in the Analysis of Metals (STP 238), 60 pp., 2000 copies.
Symposium on Application of Soil Testing in Highway Design and Construction (STP 239), 131 pp., 2000 copies.

Symposium on Effect of Water on Bituminous Paving Mixtures (STP 240), 102 pp., 2000 copies.

Symposium on Advances in Electron Metallography (STP 245), 126 pp., 2000 copies.

Symposium on Cleaning Electronic Device Components and Materials (STP 246), 217 pp., 2500 copies.

Report on Properties of Cast Iron at Elevated Temperatures (STP 248), 96 pp., 2000 copies.

Summary of Proceedings, 42 pp., 9500 copies.

Gillett Lecture, 22 pp., 3000 copies.

Special Publications in Prospect:

Index to the X-Ray Powder Data File (STP 48), 700 pp., 1000 copies.

X-Ray Powder Data File, 700 sets 3 by 5 white cards, 500 sets Keysort 4 by 6 cards (ninth set).

Cetane Manual for Rating Diesel Fuels, 110 pp., 1500 copies.

Index to Literature on Spectrochemical Analysis 1950–1955 (STP 41-D), 250 pp., 2500 copies.

Bibliography and Abstracts on Electrical Contacts (STP 56-M), 50 pp., 1500 copies.

Symposium on Particle Size Measurement (STP 234), 254 pp., 2500 copies.

Symposium on Paper and Paper Products—New Developments with Accompanying Requirements for New Testing Methods (STP 241), 92 pp., 2000 copies.

Symposium on Bulk Sampling (STP 242), 70 pp., 2000 copies.

Symposium on Materials Research Frontiers (STP 243), 54 pp., 2500 copies.

Symposium on Stability of Distillate Fuel Oils (STP 244), 80 pp., 2000 copies.

Symposium on Plastics Testing and Standardization (STP 247), 150 pp., 2500 copies.

Symposium on Bulk Quantity Measurement (STP 249), 48 pp., 2500 copies.

Symposium on Instrumentation in Atmospheric Analysis (STP 250), 64 pp., 2500 copies.

Marburg Lecture, 40 pp., 3000 copies.

The Program in Prospect for the 1959 Annual Meeting Includes the Following Symposiums:

Symposium on Education in Materials.

Symposium on Time Rates of Loading in Soil Testing.

Symposium on Methods of Test for Design of Bituminous Paving Mixtures.

Symposium on Atterberg Limits.

Symposium on Practical and Statistical Significance of Tests and Properties of Bituminous Binders.

Symposium on Spectroscopic Excitation Sources.

Symposium on Microscopy.

Symposium on Identification of Water-Formed Deposits.

Symposium on Visual Aids for Standardizing and Communicating Product Appearance.

Respectfully submitted on behalf of the committee,

R. E. HESS,
Chairman

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

The Administrative Committee on District Activities held one meeting during the year. The majority of its work was accomplished by correspondence. At the meeting in June 1958, H. F. Beeghly, H. P. Hagedorn, A. H. Kidder, and Robert Sergeson were added to the committee. The administrative committee sponsored its annual breakfast on June 25, 1958, for District Council Officers. Once again it proved to be a valuable chance to exchange information.

During the year every ASTM District held at least one meeting. The total for the year was 37, an all-time high, including two special meetings at Seattle and Richland, Wash. Of these meetings, 29 were jointly sponsored with other technical organizations.

The Rocky Mountain and Central New York Districts were officially formed and elections conducted by the Administrative Committee on District Activities. The inaugural meetings were held in Salt Lake City, Utah, and Troy, N. Y., respectively. President Woods and Executive Secretary R. J. Painter were present on both occasions.

The Administrative Committee on District Activities has encouraged District Councils to appoint membership chairmen and local public relations representatives. In such districts as have complied with these requests, noticeable achievements in membership and news reports have been apparent.

The Student Award Program has been extended, with 12 districts now presenting over 700 awards. This year the St. Louis District has participated for the first time and the Cleveland District reinstated its program. In addition, the Washington District has a program under study.

In connection with the national meetings, the Pittsburgh District, through a committee on arrangements, was host to Committee Week held in February 1959. Two Industrial Luncheons for building materials and metals were held, together with a dinner featuring an address by J. Warner, President of Carnegie Institute of Technology.

The Philadelphia District will be host to the Annual Meeting to be held June 21–26, 1959, at Atlantic City, N. J. The arrangements will include a Ladies Program and entertainment for the Annual Dinner.

Several districts, including the Southwest, Southern California, Northern California, Central New York, Washington, and Philadelphia, cooperated in local Science Fairs and observance of National Engineers Week, thus increasing the awareness of other societies of the purposes of ASTM.

Respectfully submitted on behalf of the committee,

M. N. Clair,
Chairman.

A. L. Batik,
Secretary.

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

The Administrative Committee on Research held two meetings during 1958: on June 22 in Boston, Mass., and on December 2 at ASTM Headquarters.

There are now eight members of the committee. R. C. Alden and B. W. Gonser accepted reappointment for three years, H. K. Nason declined reappointment, and W. O. Baker and E. I. Shobert II became new members of the committee.

The research fund gained substantially during the year and now amounts to over $96,000. A contribution of $2200 to Committee E-7 on Nondestructive Testing was authorized to help pay for reference radiographs for aluminum and magnesium castings and for reference radiographs for use in inspecting heavy cylindrical steel castings. This project is being financed in cooperation with several other organizations. In making this contribution, a request was made that the project be conducted to add to the knowledge of nondestructive testing and, if possible, results of interest be made available in publishable form. Although the research fund, and income from it, is not sufficient to support sizeable researches, the technical committees of ASTM have been informed again of the availability of this financial aid for worthy projects.

As in former years, many of the research projects of ASTM technical committees were reviewed, including those working cooperatively with other societies. A review of research activities of technical committees is being assembled slowly to record work in progress and results, where possible, of current and recent ASTM research projects. To assure that loss of research results is minimized, strong encouragement has been given to the establishment of a repository for technical reports at ASTM Headquarters.

The symposium on "Materials Research Frontiers" sponsored at the meeting in Boston in collaboration with the New England District Council has been published as *ASTM STP No. 243.*

As a regular contribution to the ASTM BULLETIN, short articles by various members of the Administrative Committee were prepared for publication under "ACR Notes."

The Administrative Committee believes that all of the Society's work on outdoor deterioration of materials should be coordinated and promoted. It accordingly is making inquiry of the Advisory Committee on Corrosion as to whether it might extend its purview to cover outdoor deterioration of materials generally. ASTM activities in the deterioration field should be in cooperation with the Prevention of Deterioration Center of the National Research Council.

A plan was approved to explore the feasibility of ASTM joint sponsorship of scientific conferences relating to fundamentals of materials and measurements. Also, a suggestion that the technical committees be encouraged to increase participation of materials scientists and academic people in committee activities was approved.

During this year much attention has been given to planning ahead and to working out a more clear-cut and effective field of activity for the Administrative Committee on Research. As one important step in this direction, a research forum on "The Place of Research in ASTM" is being planned for the 1959 Annual Meeting. This forum is particularly directed to technical committee officers. It should aid greatly in informing the technical committee management of the problems of the Administrative Committee on Research and of the thoughts about research activities by the officers of ASTM. In turn, the desires and expectations of the technical committee officers may help bring a closer understanding of mutual problems and how they can best be solved.

Respectfully submitted on behalf of the committee,

BRUCE W. GONSER,
Chairman.

FRANK Y. SPEIGHT,
Secretary.

APPENDIX VI

REPORT OF ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

One meeting was held during the past year: at Boston, Mass., on June 23, 1958.

Situations were discussed where conventional tests do not give realistic answers. Under conditions of supersonic flight, aircraft panels sometimes fail in an unexplained manner. The term "sonic fatigue" is often used to designate this behavior. Component failures in missiles are also difficult to rationalize in certain instances. Studies of "random vibrations" and "white noise" (vibration comprising a wide range of frequencies) are in progress at many locations; it is important that the behavior of materials under such conditions be understood and that suitable testing means be developed. The Administrative Committee feels that it has a duty to bring the situation to the attention of the Society and is proposing to do this by developing an appropriate session or symposium for the June 1960 meeting.

Brittle fracture continues to be an important problem. The committee sponsored a session at the Boston meeting on "Crack Propagation."[1] The papers by Irvin, by Hartbower and Orner, and by Lubahn and Yukawa, together with the discussions, represent a significant contribution to knowledge relating to brittle fracture, another problem in which the answer is not given by conventional tests.

The committee feels that it has concentrated its discussions too heavily in the metals field and is considering means of covering such areas as housing materials, fibers, electronic elements, and so on.

The committee has lost a valuable member in the death of Sam Gordon of Battelle Memorial Institute. His advice on aircraft problems has been most helpful.

Respectfully submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

J. W. CAUM,
Secretary.

---

[1] The papers presented at this session appear in *Proceedings*, Am. Soc. Testing Mats., Vol. 58 (1958).

REPORT OF COMMITTEE A-1
ON
STEEL*

Several extremely significant developments have occurred in the standardization program of Committee A-1 during the past year. Basic oxygen steel production has become of increasing importance in this country. As a result Committee A-1 acted to permit this new type of steel to be furnished under many of its specifications. Structural steel products affected include steel for bridges and buildings (A 7), steel for welding (A 373), steel for locomotives and cars (A 113), steel for ships (A 131), low and intermediate tensile strength plates (A 283), and rivet steel (A 141). In the concrete reinforcement area, products included billet-steel bars (A 15), cold-drawn wire (A 82), special large sizes 14S and 18S deformed billet-steel bars (A 408), and high-strength billet-steel bars (A 431). Provisions for steel made to this process were added to the general requirements for steel bars (A 29), to hot-rolled carbon steel bars (A 107), and to cold-finished carbon steel bars (A 108). All of the Committee A-1 specifications covering sheet and strip products now permit this process, including Specifications A 109, A 245, A 303, A 365, A 366, A 374, A 375, A 414, A 415, A 424, and A 425. Pipe products covered by Specifications A 53 and A 120 were affected by this change last year. Presently being considered are boiler tubes (A 83 and A 178), electric resistance-welded pipe (A 135), heat exchanger and condenser tubes (A 214), and pipe piles (A 252).

With its 1958 Report, Committee A-1 recommended for publication Tentative Specifications for Uncoated Stress-Relieved Wire for Prestressed Concrete (**A 421 – 58 T**)[1] as a follow-up document to the Tentative Specifications for Uncoated Seven-Wire Stress-Relieved Strand for Prestressed Concrete (**A 416 – 57 T**).[1] The heavy construction program still continues to be of great industrial importance, and as a result during the past year the Tentative Specifications for High-Strength Billet-Steel Bars for Concrete Reinforcement (**A 431 – 58 T**)[1] were agreed upon and published. With this report the committee is recommending to the Society proposed Tentative Specifications for Deformed Billet Steel Bars for Concrete Reinforcement with 60,000 psi Minimum Yield Point.[2] In addition, revisions are proposed for rail-steel bars for concrete reinforcement (A 16), for cable for prestressed concrete (A 416), for wire for prestressed concrete (A 421), and for high-strength billet-steel bars (A 431).[3]

Early in 1958 the Society was asked by the Boiler and Pressure Vessel Committee of The American Society of Mechanical Engineers to set minimum carbon limitations for the regular 300 series of austenitic steels. Since this was a problem common to specifications written by both

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] 1958 Book of ASTM Standards, Part 1.

[2] The new tentative specification appears in the 1959 Supplement to Book of ASTM Standards, Part 1.

[3] The revised specifications appear in the 1959 Supplement to Book of ASTM Standards, Part 1.

Committee A-1 and Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys, a joint group was established. The report of this group was forwarded to the appropriate A-1 and A-10 subcommittees for action early in January, 1959. The revisions proposed in the Appendix for Tentative Specifications A 213, A 249, and A 312, and for Standard Specifications A 271 are the first actions as a result of the joint task group report.[3] New grades identified by the suffix "H" will be added to the specifications, with chemical compositions slightly different from the regular 300 series. Solution treating temperatures will be specified for the new grades.

There were many other actions taken during the year and referred to the Administrative Committee on Standards for approval. The specifications affected are listed later in the report.

The committee also approved during the year a suggestion that a special compilation of standards covering coated and uncoated steel sheet and strip products be published. Committee A-5 and Committee A-1 would jointly sponsor this publication.[4]

Subcommittee XXIV on Heat Treatment of Steel was discharged at the January 16, 1959 meeting of the Executive Committee. This has been a dormant group for many years, with the subject of heat treatments being decided in the individual product subcommittees.

At the same meeting decision was made to organize a subcommittee covering wire rope products in response to a request from the Wire Rope Technical Board that ASTM sponsor specifications for these products. The Wire Rope Technical Board is an association of American wire rope engineers, having representation from practically all the manufacturers of wire rope in this country.

Meetings.—Committee A-1 met on June 25, 1958, in Boston, Mass., in conjunction with the 1958 Annual Meeting of the Society. On June 22, 23, and 24 twenty-four subcommittees, sections, and task groups met to prepare reports for committee action.

Committee A-1 also held a very successful series of meetings in Chicago, Ill., on January 14, 15, and 16, 1959. Committee A-1 met on January 16, with twenty-six subcommittees, sections, and task forces meeting on January 14 and 15. On the evening of January 14 the Chicago District Council sponsored a dinner with A. A. Bates, Vice-President of Research and Development, Portland Cement Assn., and Vice-President of the Society speaking on "The Impending Energy Revolution."

Personnel.—The committee consists of 333 voting members, including 146 producers, 139 consumers, and 48 general interest members.

At the January 16, 1959 meeting, L. H. Winkler, Bethlehem Steel Co., was made an Honorary Member of Committee A-1. Mr. Winkler has been a member of Committee A-1 since 1924, and was elected an Honorary Member of the Society in 1954. Many of the accomplishments of Committee A-1 are due directly to the counsel and help of Mr. Winkler during his 35 years of service.

It is with deep regret that the committee records the deaths of several previous long-time members of Committee A-1. T. G. Stitt, retired in 1953 from the Pittsburgh Steel Co., was a member of Committee A-1 for 30 years and served as chairman of Subcommittee IX on Steel Tubing and Pipe from 1934 to 1953. During his term of office, a number of ASTM specifications for steel tubular products became available and have been of widespread service to industry. Mr. Stitt was elected an Honorary Member of Committee A-1 in 1953 and received the ASTM Award of Merit in 1950.

---

[4] 1959 Compilation of ASTM Standards on Coated and Uncoated Iron and Steel Sheet and Strip

O. B. Schultz, retired Chief Metallurgist and Manager of Inspection of the Construction Equipment Div., Baldwin-Lima-Hamilton Corp., held membership in Committee A-1 from 1927 to 1954, serving on Subcommittees VI, VIII, IX, and XI. S. E. Printz, retired Engineer of Tests, Lehigh Railroad Co., served as representative of his company on Committee A-1 from 1935 to 1954. C. P. Van Gundy represented the Baltimore & Ohio Railroad on the steel committee from 1925 to 1938 and after retirement retained a personal membership until his death in 1958. This represented 33 years of continuous service, including membership in Subcommittees IV, VII, and XI.

J. T. MacKenzie, retired chemist and metallurgist, American Cast Iron Pipe Co., was widely known here and abroad for his technical papers on cast iron. Mr. MacKenzie was elected an Honorary Member of ASTM in 1956. He served on Committee A-1 from 1943 to 1958, being a member of Subcommittees VIII and XXII.

With the formation of Subcommittee XXVI on Steel Bolting in 1947, many new members were brought into Committee A-1. One of these men was F. A. McCoy, Chief Metallurgist, Sheffield Steel, Division of Armco Steel Corp. Mr. McCoy died August 25, 1958.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On September 9, 1958, the Administrative Committee on Standards, acting for the Society, accepted the following recommendations of Committee A-1:

New Tentative Specifications for:

Austenitic Steel Forged or Bored Pipe for High-Temperature Service (A 430 – 58 T), and

High-Strength Billet Steel Bars for Concrete Reinforcement (A 431 – 58 T).

Revision of Tentative Specifications for:

Billet-Steel Bars for Concrete Reinforcement (A 15 – 57 T),

Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 – 55 T),

Cold-Rolled Carbon Steel Strip (A 109 – 58 T),

Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 57 T),

Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 56 T),

Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 57 T),

Alloy Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217 – 57 T),

Factory-Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings (A 234 – 58 T),

Flat-Rolled Carbon Steel Sheets of Structural Quality (A 245 – 57 T),

Carbon and Alloy Steel Forgings for Turbine Generator Rotors and Shafts (A 292 – 57 T),

Carbon and Alloy Steel Forgings for Turbine Rotors and Shafts (A 293 – 57 T),

Hot-Rolled Carbon Steel Strip of Structural Quality (A 303 – 58 T),

Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 –58 T),

Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 57 T),

Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 – 57 T),

Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 – 57 T),

Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (A 352 – 58 T),

Cold-Rolled Carbon Steel Sheets, Special Killed for Miscellaneous Drawn or Severely Formed Parts (A 365 – 57 T),

Cold-Rolled Carbon Steel Sheets, Commercial Quality (A 366 – 57 T),

High Strength Low Alloy Cold-Rolled Steel Sheets and Strip (A 374 – 54 T),

High Strength Low Alloy Hot-Rolled Steel Sheets and Strip (A 375 – 54 T),

Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 – 58 T),

Special Large Size Deformed Billet-Steel Bars for Concrete Reinforcement (A 408 – 57 T),

Carbon Steel Sheets of Flange and Firebox Qualities (A 414 – 57 T),

Hot-Rolled Carbon Steel Sheets, Commercial Quality (A 415 – 57 T),
Steel Sheets for Porcelain Enameling (A 424 – 58 T), and
Hot-Rolled Carbon Steel Strip, Commercial Quality (A 425 – 58 T).

Revision and Reversion to Tentative of Standard Specifications for:

Cold-Drawn Steel Wire for Concrete Reinforcement (A 82 – 34),
Carbon Steel Blooms, Billets, and Slabs for Forgings (A 273 – 54), and
Alloy Steel Blooms, Billets, and Slabs for Forgings (A 274 – 54).

The new and revised specifications appear in the 1958 Book of ASTM Standards, Part 1.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 4 new tentatives and is recommending the revision of 28 tentatives and 6 standards, as well as a tentative revision of 1 standard. One tentative is being adopted as standard without change, and two tentatives are being withdrawn.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.[5]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Because of the many projects under way in the 20 subcommittees it is not feasible to mention all of the items under consideration. Only highlights of the current activities are covered in the following paragraphs:

During the task last year of drawing up requirements for the Specifications for Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service (A 426 – 58 T)[1] in Subcommittee XXII, a need was quite evident for similar specifications covering the austenitic grades of centrifugally cast pipe. This need was documented and presented to the Joint Coordinating Committee of Committees A-1, A-9, A-10, B-2, B-4, and F-1. It was agreed that the same task group would draft requirements for the proposed new specifications and present the draft for joint action of Committees A-1 and A-10.

Transition temperatures of steel products are appearing in specification requirements. In Subcommittee VI revised tables of mechanical requirements are being considered for forged turbine and turbine generator rotors and shafts (A 292 and A 293), including specified transition temperatures. This necessitates standard methods for determining transition temperature. A task group is considering such a method utilizing the V-notch Charpy impact test. Also in Subcommittee XI specifications for carbon steel plates with improved transition temperature will be patterned after a proposed case of the ASME Boiler and Pressure Vessel Code.

In answer to a request from the Bridge Committee of the American Welding Society, Subcommittee II is investigating requirements for a weldable type low-alloy structural steel. This could become a new specification or result in revisions to the Specifications for High-Strength Low-Alloy Structural Steel (**A 242 – 55**).[1]

Two quite important tasks are under way on bolting. An extensive revision of the Specification for Galvanized Steel Transmission Tower Bolts (**A 394 – 55 T**)[1] is being considered by Subcommittee XXVI. In Subcommittee XXII proposed specifications have been drafted for alloy steel turbine type bolting material specially heat treated for high-temperature service.

---

[5] See p. 116.

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The last subcommittee to be organized by Committee A-1 was Subcommittee XXVII on Steel Chain in 1954. The Specifications for Alloy Steel Chain (**A 391 – 58**)[1] and for Carbon Steel Chain (**A 413 – 57 T**)[1] were the initial efforts of the new subcommittee. Specification A 391 covers heat-treated alloy steel chain for slinging, hoisting, and load binding purposes; Specification A 413 chain is intended for railroad cars, construction, load binding, industrial uses, and purposes other than overhead load lifting. Presently under development are three more specifications for sprocket wheel chain, conveyor chain, and case carburized chain.

The Methods and Definitions for Mechanical Testing of Steel Products (**A 370 – 54 T**)[7] are extensively used and comprise an essential reference in many of the Committee A-1 product specifications. Over the past five years many comments and suggestions for changes have been received by Subcommittee XIII, some of which have been referred to this subcommittee by the other product subcommittees. An extensive revision of Methods A 370 is under way, taking into account all of the comments and suggestions received.

This report has been submitted to letter ballot of the committee, which consists of 333 members; 224 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. J. KANTER,
Chairman.

H. L. FRY,
Secretary.

---

[7] 1958 Book of ASTM Standards, Part 3.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Alloy-Steel Turbine Type Bolting Material Specially Heat Treated for High-Temperature Service (A 437 – 59 T), and
High-Strength Structural Steel (A 440 – 59 T).

Revision of Tentative Specification for:

Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 58 T),
Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 58 T),
Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 59 T),
Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 58 T),
Alloy-Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217 – 58 T),
Carbon and Alloy-Steel Forgings for Turbine Generator Rotors and Shafts (A 292 – 58 T),
Carbon and Alloy-Steel Forgings for Turbine Rotors and Shafts (A 293 – 58 T),
Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 58 T),
Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 58 T),
Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 – 55 T),
Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 – 58 T),
Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 – 58 T),

Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (A 352 – 58 T),

Seamless Austenitic Steel Pipe for High-Temperature Central-Station Service (A 376 – 56 T),

Alloy Steel Castings Specially Heat Treated for Pressure Containing Parts Suitable for High-Temperature Service (A 389 – 57 T),

Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 – 58a T),

Factory-Made Wrought Carbon Steel and Alloy Steel Welding Fittings of Seamless or Welded Construction for Low Temperature Service (A 420 – 58 T), and

Austenitic Steel Forged and Bored Pipe for High-Temperature Service (A 430 – 58 T).

Withdrawal of Standard Specifications for:

Heat-Treated Carbon-Steel Bolting Material (A 261 – 56).

The new Tentative Specification A 440 – 59 T was accepted by the Standards Committee on November 11, 1959; the other recommendations were accepted on September 15, 1959. The new and revised tentative specifications appear in the 1959 Supplement to the Book of ASTM Standards, Part 1.

APPENDIX

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix, recommendations are given affecting certain specifications, both standard and tentative, covering various steel products. The specifications under revision appear in their present form in the 1958 Book of ASTM Standards, Part 1.

NEW TENTATIVES

The committee recommends for publication as tentative the following method and specifications as appended hereto:[1]

Tentative Method and Specification for:

Ultrasonic Testing and Inspection of Steel Plates of Firebox and Higher Quality.

Tentative Specifications for:

Deformed Billet Steel Bars for Concrete Reinforcement with 60,000 psi Minimum Yield Point,

Leaded Carbon Steel Plates of Flange and Firebox Qualities for Fusion Welded Boilers and Other Pressure Vessels, and

Quenched and Tempered Alloy Steel Bars, Hot-Rolled or Cold-Finished.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Rail-Steel Bars for Concrete Reinforcement (A 16 – 57 T):

Section 1.—Revise to read as follows:

1. (*a*) These specifications cover two grades of rail steel concrete reinforcement bars: namely, regular and special.

(*b*) Regular grade bars are available in two classes: namely, plain and deformed.

(*c*) Special grade bars are available only as deformed bars. A deformed bar in either grade is defined as a bar which conforms to the latest issue of the Specifications for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (ASTM Designation: A 305). The standard sizes of deformed bars with number designations shall be those listed in Table I. The standard sizes of plain bars shall be designated by their nominal diameters.

Table II.—Over the second and third columns, add a heading to read "Regular Grade." Add a fourth column with the following values:

	Special Grade
	Deformed Bars
Tensile strength, min, psi	90 000
Yield point, min, psi	60 000
Elongation in 8 in., min, per cent	$\frac{1\ 000\ 000^{b}}{\text{tens str}}$

Section 3 (d).—Add to the end of this paragraph "to a minimum of 5 per cent."

Section 4.—Delete this section covering "Bending Properties" and renumber subsequent sections accordingly.

Section 5.—Renumber as Section 4 and in the first sentence delete the words "and bend test."

Section 6.—Renumber as Section 5 and delete the words "and one bend test."

Tentative Specification for General Requirements for Hot-Rolled and Cold-Finished Carbon and Alloy Steel Bars (A 29 – 58 T):

Table II.—Add additional check analysis requirements for nickel and new

requirements for tungsten and aluminum as follows:

---

[1] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 1.

Elements	Limit, or Maximum of Specified Range, per cent	Permissible Variations Over Maximum Limit or Under Minimum Limit, per cent
Nickel.......	Over 5.30 to 10.00, incl....	0.10
Tungsten....	1.00 and under..	0.04
	Over 1.00 to 4.00, incl..........	0.08
Aluminum...	0.10 and under..	0.03
	Over 0.10 to 0.20, incl..........	0.04
	Over 0.20 to 0.30, incl..........	0.05
	Over 0.30 to 0.80, incl..........	0.07
	Over 0.80 to 1.80, incl..........	0.10

Tentative Specifications for Welded and Seamless Steel Pipe (A 53 – 58a T):

Section 1 (a).—In the footnote referenced at the end of Item *(2)* in this section, delete the word "seamless."

Tentative Specifications for Seamless Steel Boiler Tubes (A 83 – 58 T):

Section 2.—Revise the first sentence to read "The material for seamless steel tubes shall be made by one or more of the following processes: open hearth, basic oxygen, or electric furnace."

Tentative Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 – 58 T):

Section 8.—In the second sentence change the word "drillings" to read "samples." Omit the words "turnings may be taken." This action will recognize spectrographic analysis.

Section 15.—Revise to read as follows:

> 15. Identification marks consisting of the manufacturer's symbol or name, designation of service rating, the designation A 105—I or A 105—II to indicate the grade of material, and the size shall be legibly stamped on each forging in accordance with the Standard Marking System for Valves, Fittings, Flanges, and Unions (SP-25-1958) of the Manufacturer's Standardization Society of the Valve and Fittings Industry, and in such position as not to injure the usefulness of the forging.

Tentative Specification for Hot-Rolled Carbon Steel Bars (A 107 – 58 T):

Section 1.—In Paragraph *(a)* add the following sentences to the definition of merchant quality: "It is generally not available in rounds, squares, and hexagons 3 in. and over or other bar sections weighing 40.8 lb per ft and over. In addition, merchant quality is not available in compositions specifying over 0.50 per cent carbon or 0.60 per cent maximum manganese, resulfurized steels, or other special purpose steels."

Tentative Specification for Cold-Rolled Carbon Steel Strip (A 109 – 58 T):

Table III.—Delete Footnote *a* covering the forming tool edge for the bend test of half hard material.

Tentative Specifications for Electric-Resistance-Welded Steel Pipe (A 135 – 58 T):

Section 1.—At the end of the second sentence add a reference to a footnote which will read "This provision is not intended to prohibit the cold bending of grade B pipe."

Section 2.—Revise to read "The steel shall be made by one or more of the following processes: open hearth, basic oxygen, or electric furnace."

Tentative Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 58 T):

Section 2.—Revise to read as follows: "The steel or iron shall be made by one or more of the following processes:

open hearth, basic oxygen, or electric furnace."

Tentative Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for General Service (A 181 - 58 T):

Section 7.—Revise as indicated above for Section 8 in Specifications A 105.

Section 14.—Revise as indicated above for Section 15 in Specifications A 105, but use the designations "A 181—I" and "A 181—II."

Tentative Specifications for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 - 58 T):

Section 7.—In the second sentence change the word "drillings" to read "a sample."

Section 8.—Revise as indicated above for Section 8 in Specifications A 105.

Tentative Specifications for Seamless Alloy Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 - 58 T):

Section 1 (b).—Revise to read "Grades TP 304 H, TP 316 H, TP 321 H, TP 347 H, and TP 348 H are modifications of grades TP 304, TP 316, TP 321, TP 347, and TP 348, and are intended for high-temperature service such as for superheaters and reheaters."

Section 4.—Revise Paragraph (*a*) to read "All material except grades TP 304 H, TP 316 H, TP 321 H, TP 347 H, and TP 348 H shall be furnished in the . . . ."

Add new Paragraph (*d*) to read as follows:

> (*d*) All H grades shall be furnished in the solution treated condition. If cold working is involved in processing, the minimum solution treating temperature for grades TP 347 H and TP 348 H shall be made 2000 F and for grades TP 304 H and TP 316 H, 1800 F. If the H grade is hot rolled, the minimum solution treatment for grades TP 321 H, TP 347 H, and TP 348 H shall be 1925 F, and for grades TP 304 H and TP 316 H, 1800 F.

Table II.—Add new grades TP 304 H, TP 316 H, TP 347 H, and TP 348 H with compositions similar to the regular grades of the same number, except that the carbon contents shall be listed as "0.04 to 0.10" per cent. For grades TP 347 H and TP 348 H the columbium plus tantalum content shall be 8 × carbon minimum, 1.0 per cent maximum.

Section 21 (a).—Revise the last sentence to read "For grades TP 304 H, TP 316 H, TP 321 H, TP 347 H, and TP 348 H, the marking shall also include the heat number and the heat treatment lot identification."

Tentative Specifications for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 58 T):

Section 2.—Revise to read "The steel shall be made by one or more of the following processes: open hearth, basic oxygen, or electric furnace."

Tentative Specifications for Factory-Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings (A 234 - 58a T):

Section 4.—Add a new Paragraph (*d*) to read "In fittings of welded construction, the alloy content (chromium and molybdenum) of the deposited weld metal shall conform to that required for the base material."

Section 5.—Revise to read "An analysis of the base metal and the weld metal may be made by the purchaser from one or more fittings in each lot.[4] The chemical composition thus determined shall conform to the requirements of Section 4.

Tentative Specifications for Welded Austenitic Stainless Steel Boiler, Superheater, Hest Exchanger, and Condenser Tubes (A 249 - 58 T):

Section 1.—Add a new Paragraph (*b*) to read as indicated above for Section

1 (*b*) in Specifications A 213, relettering subsequent paragraphs accordingly.

Section 4.—Add a new Paragraph (*c*) to read as follows:

> (*c*) All H grades shall be furnished in the solution treated condition. If cold working is involved in processing, the minimum solution treating temperature for grades TP 321 H, TP 347 H, and TP 348 H shall be 2000 F and for grades TP 304 H and TP 316 H, 1800 F. If the H grade is hot rolled, the minimum solution treating temperatures for grades TP 321 H, TP 347 H, and TP 348 H shall be 1925 F, and for grades TP 304 H and TP 316 H, 1800 F.

Table I.—Add grades TP 304 H, TP 316 H, TP 347 H, and TP 348 H with compositions similar to the regular grades of the same number, except that the carbon contents shall be "0.04 to 0.10" per cent. For grades TP 347 H and TP 348 H the columbium plus tantalum content shall be 8 × carbon minimum, **1.0** per cent maximum.

Section 23 (*a*).—Add a sentence to read as indicated above for the last sentence of Section 21 (*a*) in Specifications A 213.

Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 58 T):

Section 1.—Add a note after Paragraph (*b*) to read "NOTE.—The grades of austenitic stainless steel pipe furnished in accordance with these specifications have been found suitable for low temperature service down to −325 F, where Charpy notched bar impact values of 15 ft-lb, minimum, are required, and need not be impact tested."

Add a new Paragraph (*c*) to read as indicated above for Section 1(*b*) in Specifications A 213, relettering subsequent paragraphs accordingly.

Section 5.—Revise Paragraph (*a*) to read "All material except grades TP 304 H, TP 316 H, TP 321 H, TP 347 H, and TP 348 H shall be furnished the . . . . ."

Add a new Paragraph (*c*) to read as indicated above for Section 4 in Specifications A 249.

Table I.—Add new grades TP 304 H, TP 316 H, TP 321 H, TP 347 H, and TP 348 H with compositions similar to the regular grades of the same number, except that the carbon contents shall be "0.04 to 0.10" per cent. For grades TP 347 H and TP 348 H the columbium plus tantalum content shall be 8 × carbon minimum, 1.0 per cent maximum; and for grade TP 321 H the titanium content shall be 4 × carbon minimum, 0.60 per cent maximum.

Table II.—Add a footnote, referenced after the requirement for minimum tensile strength, to read "For grade TP 304, in sizes of 8 in. nominal diameter and larger and schedule 140 and heavier, the minimum tensile strength shall be 70,000 psi."

Section 21.—Add a new fourth sentence to read as indicated above for the last sentence of Section 21 (*a*) in Specifications A 213.

Tentative Specifications for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 58 T):

Section 9 (*a*).—In the first sentence, delete the impact test requirement for grade B 8 F.

Table III.—Delete the reference to grade B 8 F for acceptance test temperature for impact test.

Section 14.—Add a new Paragraph (*d*) to read "If washers are used under nuts, they shall be of forged or rolled steel. All washers shall be free from injurious defects and shall have a workmanlike finish."

Tentative Specification for Quenched and Tempered Carbon Steel Bars (A 321 – 57 T):

Section 8.—Change the permissible maximum carbon content from the present "0.50" to read "0.55" per cent.

TABLE I.—TENSILE REQUIREMENTS.
(Revision of Table I in Specification A 321 – 57 T)

Diameter or Distance Between Parallel Faces, in.	Yield Point, min, psi	Tensile Strength, min, psi	Elongation in 2 in., min, per cent	Reduction of Area, min, per cent
1 and under....	75 000	110 000	18	45
Over 1 to 2½, incl..........	70 000	105 000	18	45
Over 2½ to 4, incl..........	65 000	95 000	18	45
Over 4 to 6, incl..........	60 000	90 000	18	40
Over 6 to 9½, incl..........	50 000	85 000	18	35

Table I.—Revise to read as shown in the accompanying Table I.

Tentative Specifications for Seamless and Welded Steel Tubes for Low-Temperature Service (A 334 – 58 T):

Section 13 (c).—In the last sentence change the impact specimen transfer time from the present "6" to "5" seconds to conform to the requirements of the Methods and Definitions for the Mechanical Testing of Steel Products (ASTM Designation: A 370).

Section 16 (a).—Revise the last part of the third sentence by adding the italicized words ". . . . shall be taken from rounds, tubes, *slabs, or strip* before reducing to final size."

Tentative Specifications for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 – 55 T):

Section 1 (b).—Revise the first sentence to read "Twelve grades of ferritic steels[4] are covered."

Table I.—Delete grades P 3 and P 3b.

Tentative Specifications for Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 – 58 T):

Section 8.—Revise as indicated above for Section 8 in Specifications A 105.

Tentative Specifications for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts Specially Heat Treated for High-Temperature Service (A 404 – 57 T):

Section 7.—Revise as indicated above for Section 7 in Specifications A 182.

Section 8.—Revise as indicated above for Section 8 in Specifications A 105.

Tentative Specification for High-Strength Structural Alloy Rivet Steel (A 406 – 57 T):

Table I.—Change the requirements for aluminum content from the present "0.10 to 0.30" and "0.08 to 0.32" for ladle and check analysis, respectively, to read "0.15 to 0.30" and "0.10 to 0.35" per cent.

Section 7.—Revise this section covering the upsetting test to read as follows:

> 7. (*a*) On bars to be manufactured into rivets by the cold heading process, specimens cut to a length of one and one fourth times the diameter of the bar shall stand hammering down cold in a longitudinal direction to a length equal to three fourths of the original diameter, without showing seams or other defects of sufficient depth which would tend to produce defects in the manufactured rivets. These specimens shall be annealed by heating to 1450 F, holding at this temperature for not less than 30 min, and cooling slowly in the furnace.
>
> (*b*) On bars to be manufactured into rivets by the hot heading process, specimens cut to a length of one and one fourth times the diameter of the bar shall stand hammering down hot in a longitudinal direction to a length equal to three fourths of the original diameter, without showing seams or other defects of sufficient depth which would tend to produce defects in the manufactured rivets. These specimens shall be heated to a temperature not in excess of 1800 F.

Section 8.—Delete Paragraph (*c*) since this is covered in the revised Section 7.

Tentative Specification for Carbon Steel Chain (A 413 – 57 T):

This specification has been extensively

---

[2] The revised tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 1.

revised and is appended hereto in its proposed revised form.[2]

Tentative Specifications for Uncoated Seven-Wire Stress-Relieved Strand for Prestressed Concrete (A 416 - 57 T):

Section 16.—Revise this section titled "Inspection" to read as follows:

16. The purchaser shall state at the time of order whether outside inspection is required or waived. If outside inspection is required, the manufacturer shall afford the inspector representing the purchaser all reasonable facilities, without charge, to satisfy him that the material is being furnished in accordance with these specifications. All tests and inspections shall be made at the place of manufacture prior to shipment, unless otherwise agreed upon at the time of purchase, and shall be so conducted as not to interfere unnecessarily with the operation of the works. If outside inspection is waived, a manufacturer's certification that the material has been tested in accordance with and meets the requirements of these specifications shall be basis of acceptance of the material.

Tentative Specifications for Uncoated Stress-Relieved Wire for Prestressed Concrete (A 421 - 58 T):

Table II.—For wire with a nominal diameter of 0.196 in., add a minimum tensile strength of 240,000 psi for type BA.

Table III.—For wire with a nominal diameter of 0.196 in., add a minimum yield strength of 192,000 psi for type BA.

Section 8.—Add the following to the end of this section:

If the fracture takes place outside of the gage length, the elongation value obtained may not be representative of the material. If the elongation so measured meets the minimum requirements specified, no further testing is indicated; but if the elongation is less than the minimum requirements, the test shall be discarded and a retest made.

Section 15.—Revise to read as indicated above for Section 16 in Specifications A 416.

Tentative Specifications for Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service (A 426 - 58 T):

Table I.—Delete grades CP 3 and CP 3b.

Tentative Specifications for High-Strength Billet Steel Bars for Concrete Reinforcement (A 431 - 58 T):

Section 8 (b).—Revise to read as follows:

8. (*b*) The yield point shall be determined by one of the following methods:

(*1*) Extension under load using dividers with an 8-in. gage length. The extension under load shall be $\frac{3}{64}$ in. and shall be determined by scribing on the specimen an 8-in. gage length, pivoting from a prick punch mark. The yield load shall be recorded when the total gage length under load becomes $8\frac{3}{64}$ in. as measured by the dividers.

(*2*) Extension under load using an autographic diagram method or an extensometer as described in Section 7 (*f*) (*2*) and (*4*) of the Methods and Definitions for Mechanical Testing of Steel Products (ASTM Designation: A 370). However, the per cent extension under load shall be 0.006 in. per in. of gage length (0.6 per cent).

Section 10.—Revise to read as follows:

10. (*a*) Tension test specimens may be either the full section of the bar as rolled or at the option of the manufacturer one of the reduced section type of tests described in the following Items (*1*) and (*2*). The sectional area used for unit stress determination of full size specimens shall be calculated from the length and weight of the test specimen (Note).

NOTE.—The area in square inches may be calculated by dividing the weight per linear inch of specimen in pounds by 0.2833, or by dividing the weight per linear foot of specimen in pounds by 3.4.

(*1*) Standard 0.505 diameter test specimen, with 2 in. gage length, as described in Fig. 6 of Methods A 370 and from a position in the bar as outlined in Table B-1 of Methods A 370.

(*2*) Specimens machined to a reduced diameter of not less than $\frac{3}{4}$ in. for a length of not less than 9 in., with an 8-in. gage length.

(*b*) The bend test specimens, when required, shall be the full section of the bar.

Table II.—Revise the heading of the last column to read "2-in. Gage Length (Fig. 6 of Methods A 370)."

Section 11 (*b*).—Delete the words "and size of bar rolled" at the end of this sentence.

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions as follows of the Standard Specifications for Steel Music Spring Wire (**A 228 – 51**):

Table I.—Extend Table I to include the following values:

Diameter, in.	Tensile Strength, psi	
	Min	Max
0.162.........	249 000	275 000
0.177.........	245 000	270 000
0.192.........	241 000	267 000
0.207.........	238 000	264 000

Add a note to Table I to read "Tensile strength values for intermediate sizes may be interpolated."

Section 9 (*a*).—Revise to read "The diameter of the wire shall not vary from that specified by more than the amount prescribed in Table II."

New Table.—Add a new Table II to read as shown in the accompanying Table II.

TABLE II.—PERMISSIBLE VARIATIONS IN DIAMETER.

(New Table II for Specifications A 228 – 51)

Specified Diameter, in.	Permissible Variations from Specified Diameter, ± in.
0.004 to 0.010.........	0.0002
0.011 to 0.028.........	0.0003
0.029 to 0.063.........	0.0004
0.064 to 0.080.........	0.0005
0.081 and over.........	0.001

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Recommended Practices for Ultrasonic Testing and Inspection of Heavy Steel Forgings (**A 388 – 55 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specification for General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 – 56):

Section 5 (*b*).—Revise to read "For plates 1½ in. and under in thickness, tension and bend test specimens shall be the full thickness of the material, and shall be machined to the form and dimensions shown in Fig. 1, or with both edges parallel."

Standard Specification for Structural Steel for Ships (A 131 – 58):

Table I.—For class C change the maximum permissible carbon content for ladle analysis from the present "0.24" to read "0.23" per cent and for check analysis from the present "0.28" to read "0.27" per cent.

Revise the present Footnote *b* by adding the words "and the McQuaid-Ehn austenite grain size is to be determined."

Add a new Footnote *c*, referenced after the maximum carbon requirement for ladle and check analysis of grade C, to read "Plates specified to be normalized may have a maximum carbon content of

---

[3] 1958 Book of ASTM Standards, Part 3.

0.24 per cent on ladle analysis and 0.28 per cent on check analysis."

Table II.—Delete all reference to yield point.

Standard Specification for High-Strength Structural Rivet Steel (A 195 – 57):

Section 7.—Revise as indicated above for Section 7 in Specification A 406.

Section 8.—Delete Paragraph (*c*) since this is covered in the revised Section 7.

Standard Specifications for Welded and Seamless Steel Pipe Piles (A 252 – 55):

Section 2.—Revise to read "The steel shall be made by one or more of the following processes: open hearth, basic oxygen, electric furnace, or acid bessemer."

Table I.—For grade 3 change the required minimum tensile strength from the present "75,000" to read "66,000" psi.

Standard Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 58):

Section 1 (*a*).—Add the following note after this paragraph: "NOTE.—The grades of austenitic stainless steel tubing furnished in accordance with these specifications have been found suitable for low-temperature service down to −325 F, where Charpy notched bar impact values of 15 ft-lb, min, are required, and need not be impact tested."

Standard Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 58):

Section 1.—Add a new Paragraph (*b*) to read as follows, relettering subsequent paragraphs accordingly:

> (*b*) Grades TP 304 H, TP 321 H, TP 347 H, and TP 348 H are modifications of grades TP 304, TP 321, TP 347, and TP 348, and are intended for high-temperature service such as for superheaters and reheaters.

Section 4.—Revise the first sentence of Paragraph (*a*) to read "All material except grades TP 304 H, TP 321 H, TP 347 H, and TP 348 H, shall be furnished in the ....."

Add a new Paragraph (*c*) to read as follows:

> (*c*) All H grades shall be furnished in the solution treated condition. If cold working is involved in processing, the minimum solution treating temperature for grades TP 321 H, TP 347 H, and TP 348 H shall be 2000 F and for grade TP 304 H shall be 1800 F. If the H grade is hot rolled, the minimum solution treatment for grades TP 321 H, TP 347 H, and TP 348 H shall be 1925 F, and for grade TP 304 H shall be 1800 F.

Table I.—Add new grades TP 304 H, TP 321 H, TP 347 H, and TP 348 H with compositions similar to the regular grades of the same number, except that the carbon contents shall be listed as "0.04 to 0.10" per cent. For grades TP 347 H and TP 348 H the columbium plus tantalum content shall be 8 × carbon minimum, 1.0 per cent maximum. For grade TP 321 H the titanium content shall be 4 × carbon minimum, 0.60 per cent maximum.

Section 19 (*a*).—Add a sentence to read "For grades TP 304 H, TP 321 H, TP 347 H, and TP 348 H, the marking shall also include the heat number and the heat treatment lot identification."

WITHDRAWAL OF TENTATIVES

The committee recommends the withdrawal of the following tentatives:

Tentative Specifications for:

Quenched and Tempered Alloy Steel Bars (**A 286 – 57 T**), and

Cold-Finished Quenched-and-Tempered Alloy Steel Bars (**A 364 – 57 T**).

These specifications will be replaced by the proposed Tentative Specification for Quenched and Tempered Alloy Steel Bars, Hot-Rolled or Cold-Finished, appended to this report.[1]

REPORT OF COMMITTEE A-3 ON CAST IRON*

Committee A-3 on Cast Iron held two meetings during the year: in Boston, Mass., June 27, 1958, and in Pittsburgh, Pa., February 5, 1959. A number of subcommittees and task groups met in conjunction with the main committee.

The committee consists of 129 members, of whom 124 are voting members; 46 are classified as producers, 45 as consumers, and 33 as general interest members. There are 2 consulting members.

During the year, the committee lost because of death the services of two longtime and valued members: V. A. Crosby and J. T. MacKenzie. Because of the death of Mr. Crosby, the chairmanship of Subcommittee 18 on Automotive Castings is open.

Mr. W. E. Gruver was appointed chairman of Subcommittee 11 on Methods of Test, and representative of Committee A-3 on Committee E-7.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee A-3 presented to the Society through the Administrative Committee on Standards a revision of the Standard Specifications for Cast Iron Culvert Pipe (**A 142 - 38**).[1] The revision was accepted by the Administrative Committee on Standards on April 13, 1959, and appears in the 1959 Supplement to Book of ASTM Standards, Part 1.

NEW TENTATIVE

The committee recommends for publication as tentative the Proposed Specification for Austenitic Gray Iron Castings as appended hereto.[2]

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as follows of the Standard Specifications for Gray Iron Castings for Pressure-Containing Parts for Temperatures up to 650 F (**A 278 - 57**).[1]

Section 3 (a).— Delete "(Notes 1, 2 and 3)."

Section 4.—In Paragraph (*c*) delete the last sentence and replace with the following: "In either case, the cooling rate of the test bar shall closely approximate that of the controlling section of the casting represented. The approximate cooling rate of the casting may be achieved by selecting a test bar with the same surface to volume ratio."

In Paragraph (*d*), in the note following the table giving the size of test bars, delete "(Notes 5 and 6)."

Figure 1.—Delete the table under the figure and replace with the following:

Test Bar	Diameter, in.	Permissible Variations, in.
A............	0.875	±0.05
B............	1.20	±0.10
C............	2.00	±0.10

Section 5.—In Paragraph (*a*) delete

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] 1958 Book of ASTM Standards, Part 1.

[2] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 1.

the reference to Notes 5 and 6. In Paragraph (*b*) delete the reference to Notes 7 and 8.

Section 6 (*b*).—Delete the reference to Note 9.

Section 8.—Revise to read as follows:

(*a*) It is the intent of these specifications to subordinate chemical composition to mechanical properties. Cast irons for services at temperatures above 450 F shall be limited to the class Nos. 40, 50, and 60 having a maximum carbon content equivalent of 3.8 per cent as calculated by the formula: carbon content equivalent equals total carbon +0.3 (silicon + phosphorus).

(*b*) Further limitations of the chemical composition of class Nos. 40, 50, and 60 shall be:

Phosphorus, max, per cent	0.25
Sulfur, max, per cent	0.12

Explanatory Notes.—Delete all Explanatory Notes.

Figures 3, 4, and 5.—Delete these figures.

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Methods of Testing Chilled and White Iron Castings (**A 360 - 55 T**)[3] because it was discovered the methods were not being used.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Foundry Pig Iron (**A 43 - 55 T**), and
Cast Nodular Iron for Pressure Containing Parts for Use at Elevated Temperatures (**A 395 - 56 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Task Group on Chemical Analysis of High-Carbon Irons (George Krumlauf, chairman) is investigating ASTM methods of sampling and chemical analyses of high-carbon irons.

Task Group on International Standards (D. E. Krause, chairman).—The task group has maintained close contact with ISO Technical Committee 25 on Cast Iron but was unable to finance representation at the ISO meeting held in June, 1958, at Harrogate, England. Because of apathy of industrial organizations there is some thought that the USA revert to observer status, but active status is being continued for the present. The task group considered and acted upon numerous matters concerning the international classification of gray irons, design of test bars and test specimens, tension testing, hardness testing, and impact testing.

The Task Group recommended to ASA that the USA cast negative votes on proposals ISO/TC25/68E on Brinell Hardness Testing and ISO/TC25/70E on Gray Cast Iron.

Task Group on Test Bars (G. A. Timmons and H. W. Lownie, Jr., cochairmen).—The Task Group, made up of members of Subcommittees VI and XI, has prepared recommendations for the adoption of new short test bars for tension testing to replace the present long ("transverse") test bars. The new bars are being incorporated into a revision of Specifications A 48. The long bars are recommended only for retention as a control test and are being covered in a new proposed recommended practice.

Task Group on Exposure of Atmospheric Test Plates (D. E. Krause, chairman) was dissolved and the project discontinued because of insufficient interest in and demand for this activity.

Subcommittee 1 on Pig Iron (H. W.

---

[3] 1958 Book of ASTM Standards, Part 3.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Stuart, chairman).—A task group reported that several years of study led to the conclusion that the present Bureau of Standards samples are satisfactory for standardization of laboratory equipment for determination of total carbon and that additional standards are not needed. However, the techniques for sampling and analysis of high-carbon irons need further work and are being studied by the Task Group on Chemical Analysis of High-Carbon Irons.

Subcommittee VI on General Castings (H. W. Lownie, Jr., chairman).—A major revision of Specifications A 48 – 56 has been drafted and is based on the recommendations of the Task Group on Test Bars. The revision calls for the deletion of long ("transverse") test bars for qualification of the irons in Specifications A 48.

Subcommittee VIII on Cast Iron Culvert Pipe (W. F. Abercrombie, chairman).—Specifications A 142 were revised to provide for alternate terms for qualification.

Subcommittee XII on Austenitic Cast Iron (Hugo Larson, chairman).—The subcommittee prepared the proposed Specification for Austenitic Gray Iron Castings referred to earlier in the report.

Subcommittee XII has been activated as a task group of Subcommittee XXIV to prepare a new specification on austenitic nodular iron.

Subcommittee XIX on Chilled and White Iron Castings (N. R. Arant, chairman).—The subcommittee recommended the withdrawal of Tentative Methods A 360 – 55 T because it was learned the methods were not being used.

The need for a new specification for wear- and abrasion-resistant castings was studied. It was concluded that the use is so specialized that a suitable general specification cannot be prepared.

The subcommittee began preparation of a new specification for chilled iron rolls for the rubber and plastic industries.

Subcommittee XX on Elevated-Temperature Properties (J. S. Vanick, chairman).—A research project sponsored by the Joint ASTM-ASME Research Committee on Effect of Temperature (Project SP-2) on the properties of cast iron at elevated temperatures is to be printed and distributed in 1959. Reports on and references to elevated-temperature work were submitted to the ASME (for which this subcommittee acts as reporter).

Subcommittee XXIV on Nodular Iron (H. W. Ruf, chairman).—A task group under the chairmanship of H. Larson has prepared and distributed drafts of a proposed new specification on austenitic nodular iron.

A task group under the chairmanship of S. Low has prepared preliminary drafts of a proposed new specification on nodular iron valves, flanges, and pipe fittings.

Another task group under the chairmanship of W. J. Sommer is working on the problem of test bars for nodular iron.

This report has been submitted to a letter ballot of the committee, which consists of 124 voting members; 105 members returned their ballots, of whom 99 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. E. EAGAN,
Chairman.

H. W. LOWNIE, JR.,
Secretary.

REPORT OF COMMITTEE A-5
ON
CORROSION OF IRON AND STEEL*

Committee A-5 on Corrosion of Iron and Steel held two meetings during the past year: on June 24, 1958, at Boston, Mass., and on February 4, 1958, at Pittsburgh, Pa.

The committee consists of 103 members, of whom 45 are classified as producers, 31 as consumers, and 27 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On September 9, 1958, the Administrative Committee on Standards, acting for the Society, accepted the following recommendations of Committee A-5:

Revision of Tentative Specifications for:

Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils, and Cut Lengths (A 93 - 58 T),

Long-Terne Iron or Steel Sheets, Coils, and Cut Lengths (A 308 - 58 T), and

1.25 oz Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets (A 361 - 58 T).

The revised specifications appear in the 1958 Book of ASTM Standards, Part 1.

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for 1.25 oz Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets (A 361 - 58 T):[1]

Section 8 (b).—Change to read as follows: "(*b*) For coils or cut length sheets coated on a continuous line, the spot test shall be made from each 30,000 lb lot or fraction thereof of each gage."

This change has been made to make the number of tests agree with the number required in other sheet specifications.

Tentative Specifications for Zinc-Coated Steel Chain-Link Fence Fabric (A 392 - 55 T):[1]

Section 10 (a).—Change the third sentence to read as follows: "Both galvanized-before-weaving and galvanized-after-weaving fabric shall be given careful visual inspection to determine the quality of the zinc coating."

This change stresses the fact that fabric galvanized before weaving is also subject to careful visual inspection. The present wording can be interpreted to mean that only fabric galvanized after weaving is subject to careful visual inspection.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

[1] 1958 Book of ASTM Standards, Part 1.

Tentative Recommended Practice for:

Safeguarding Against Warpage and Distortion During Hot-Dip Galvanizing of Steel Assemblies (**A 384 - 55 T**).[1]

Tentative Specifications for:

Zinc-Coated Low Carbon Steel Armor Wire (**A 411 - 57 T**).[1]

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Recommended Practice for Providing High Quality Zinc Coatings (Hot-Dip) on Assembled Products (A 385 - 55 T):[1]

Section 10.—Delete the sentence: "An accepted practice is to use a tap $\frac{1}{64}$ in. (0.0156 in.) oversize for all American Standard Coarse threads $\frac{1}{4}$ in. diameter and larger."

The reason for this deletion is that there is some disagreement among manufacturers concerning the size or sizes of the tap to be used. The subject is now being reviewed.

Tentative Specifications for Zinc Coating (Hot-Dip) on Assembled Steel Products (A 386 - 55 T):[1]

Section 2.—Amend this section to read: "The zinc used for the coating shall conform to the Specifications for Slab Zinc (Spelter) (ASTM Designation: B 6), and shall be at least equal to the grade designated as Prime Western."

Table I.—Delete the Note under Table I and substitute the following: "NOTE.—Several magnetic or electromagnetic types of thickness gages are commercially available and are satisfactory for inspection when properly calibrated just prior to inspection use."

Section 5.—Delete the following words in the Note: "in which the thickness of the zinc coating can be controlled."

Section 7 (a).—Add the following sentence to the end of the paragraph: "Material found to be embrittled shall be rejected."

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot vote of the Society:

Standard Specifications for Zinc-Coated (Galvanized) "Iron" Telephone and Telegraph Line Wire (A 111 - 52):[1]

Section 14 (c).—Change to read as follows:

> (*c*) Each coil shall be securely bound in four separate places, equally spaced, with at least two wrappings of galvanized wire not smaller than 0.080 in. in diameter. As an alternative, coils may be securely bound in three separate places, equally spaced, with a zinc-coated steel strap. The steel crimping seal used to fasten the strap may be either zinc-coated or painted steel. For bundles with a nominal weight less than 100 lb the steel strap shall be not less than $\frac{1}{2}$ in. wide by 0.020 in. thick. For bundles with a nominal weight of 100 lb or more the steel strap shall be not less than $\frac{5}{8}$ in. wide by 0.020 in. thick, and each binder shall be looped around several strands of wire and then passed around the coil and tightened. The outer end of each coil shall be bent back over the binding wire or strap or else tucked into the coil and shall be identified by an appropriate tag to facilitate locating the end.

The change in Section 14 (*c*) will permit straps instead of tie wires for all weights of coils. It also designates the sizes of straps to be used and calls for an appropriate tag to facilitate locating the ends of coils if the end of the wire is tucked into the coil.

Standard Specifications for Zinc-Coated (Galvanized) Steel Tie Wires (A 112 - 58):[1]

Section 1.—Change to read as follows:

1. These specifications cover zinc-coated (galvanized) steel tie wires with class A, class B, and class C coating weights, in cut lengths for use in tying zinc-coated telephone and telegraph line wire to insulators.

Section 2.—Change the information required on the order to read:

(*1*) Quantity of each size,
(*2*) Class of coating weight (Section 7),
(*3*) Wire size, diameter in inches (Section 13),
(*4*) Length required (Section 14), and
(*5*) Weight of bundles and type of packing (Section 17).

Section 3.—Change to read: "The slab zinc, when used for the coating, shall be any grade of zinc conforming to the Standard Specifications for Slab Zinc (Spelter) (ASTM Designation: B 6)."

Section 7.—Change to read: "The weight of zinc coating, in oz per sq ft of uncoated wire surface, shall be not less than that specified in Table I."

New Table.—Add a new Table I as shown in the accompanying Table I.

TABLE I.—MINIMUM WEIGHTS OF COATING.
(New Table I for Specifications A 112 - 58)

Nominal Diameter of Coated Wire, in.	Minimum Weight of Coating, oz per sq ft of Uncoated Wire Surface		
Class	A	B	C
0.109 and over	0.80	1.60	2.40
0.095 and under	0.70	1.40	2.10

Section 18.—Change to read: "To each bundle or carton shall be securely fastened a strong tag or label showing the nominal size of the zinc-coated tie wire, class of coating, gross weight, specified length, and the name or mark of the manufacturer."

The changes to Specifications A 112 consist of the addition of two classes of tie wires with heavier zinc coatings and minor changes made necessary by these additions.

Standard Specifications for Zinc (Hot-Galvanized) Coatings on Products Fabricated from Rolled, Pressed, and Forged Steel Shapes, Plates, Bars, and Strip (A 123 - 53):[1] Revise as appended hereto.[2]

The several paragraphs which have been added to Specifications A 123 are of a cautionary and instructive nature and should aid in producing good galvanizing jobs. It has been amply demonstrated that the recommended coating weight for material ¼ in. and thicker can be achieved without undue difficulty. Work of the committee has shown that several electromagnetic and magnetic type thickness gages have sufficient accuracy to make them suitable for use in determining the uniformity of zinc coatings. Their use for this purpose has therefore been suggested. The incorporation of the pivoted hammer to test the adherence of zinc coatings serves to standardize the force of the blow.

Standard Recommended Practice for Safeguarding Against Embrittlement of Hot Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A 143 - 46):[3]

Section 1.—Change "1" to "1(*a*)." Add a new Paragraph (*b*) to read as follows:

(*b*) The design of the product and the selection of the proper steel for its suitability to be fabricated and to withstand normal galvanizing operation without embrittlement is the responsibility of the fabricator. The galvanizer shall not damage the material by overpickling or by the use of excessively high temperatures in pickling or galvanizing."

---

[2] The revised specification appears in the 1959 Supplement to Book of ASTM Standards, Part 1.

[3] 1958 Book of ASTM Standards, Part 3.

Section 3.—Add after "open hearth" the words, "basic oxygen, and electric furnace" and change the word "should" to "shall." At the end of Section 3 add the following Note:

NOTE.—Susceptibility of steels to galvanizing embrittlement is also mentioned in the Appendix to the Recommended Practice for Safeguarding Against Warpage and Distortion During Hot-Dip Galvanizing of Steel Assemblies (ASTM Designation: A 384).

Section 4 (c).—In the last sentence change "should" to "shall."

Standard Specifications for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (A 153 – 53):[1] Revise as appended hereto.[2]

The changes in Specifications A 153 consist generally of the addition of certain cautionary paragraphs, the deletion of reference to the Preece test for the determination of the uniformity of zinc coatings, and the suggested substitution therefor of magnetic or electromagnetic thickness gages, and necessary changes because of the elimination of the Preece test.

Standard Specifications for Zinc-Coated (Galvanized) High Tensile Steel Telephone and Telegraph Line Wire (A 326 – 52):[1]

Section 14 (c).—Change to read as follows:

(*c*) Each coil shall be securely bound in four separate places, equally spaced, with at least two wrappings of galvanized wire not smaller than 0.080 in. in diameter. As an alternative, coils may be securely bound in three separate places, equally spaced, with a zinc-coated steel strap. The steel crimping seal used to fasten the strap may be either zinc-coated or painted steel. For bundles with a nominal weight less than 100 lb the steel strap shall be not less than ½ in. wide by 0.020 in. thick. For bundles with a nominal weight of 100 lb or more the steel strap shall be not less than ⅝ in. wide by 0.020 in. thick, and each binder shall be looped around several strands of wire and then passed around the coil and tightened. The outer end of each coil shall be bent back over the binding wire or strap or else tucked into the coil and shall be identified by an appropriate tag to facilitate locating the end.

The change in Section 14 (*c*) is for the same reason as in Specifications A 111.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Methods of Testing (O. B. Ellis, chairman) will investigate a nitric acid dip test and a copper fluoride test for the determination of the continuity of aluminum coatings. The Schikorr test will also be investigated in connection with the testing of zinc coatings. A specification for determining coating thicknesses by magnetic gages will be undertaken.

Subcommittee XI on Sheet Specifications (E. P. Beachum, chairman) recommended the revisions to Specifications A 93, A 308, and A 361 which were approved by the Administrative Committee on Standards. It also recommended the further revision to Specifications A 361 referred to earlier in this report. Progress has been made in preparing specifications for galvanized sheets of structural quality, and for galvanized sheets for culverts and underdrains. The subcommittee plans to prepare a specification for aluminum-coated steel sheets in response to increased interest in this material.

Subcommittee XII on Wire Specifications (J. F. Occasione, chairman) has revised the workmanship requirement of the Tentative Specifications A 392 and recommended its retention as a tentative specification to determine suitability of the revision and to further modify the specification with regard to the calculation of surface area (Section 7 (*c*)).

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Tentative Specifications A 411 were recommended for adoption as standard. Revisions were made for immediate adoption as standard to Specifications A 111, A 112, and A 326. A draft specification is being prepared on copper-covered steel guy strand.

A survey on the status of aluminum coating of wire was conducted and preparation of specifications for various wire products will be undertaken. Work is continuing on the preparation of a draft of a specification for zinc coated flat armor tape.

Subcommittee XIII on Hardware Specifications (B. J. Barmack, chairman) reviewed Tentative Specifications A 384, A 385, and A 386, and recommended their adoption as standard. Specifications A 123 were revised to show the average weight of coating of 2.3 oz per sq ft of surface and a minimum of 2.0 oz for $\frac{1}{4}$ in. and thicker steel. The values of 2.0 oz average and 1.8 oz minimum were retained for $\frac{1}{8}$ and $\frac{3}{16}$ in. material.

Specifications A 153 were reviewed. The table of coating weights was not revised pending the development of a suitable sampling method by Committee E-11. It can be shown by the use of statistical methods that the average weights could be raised if a larger number of specimens is used in the sampling procedure. The use of magnetic thickness gages is recommended to replace the obsolete Preece test.

Recommended Practice A 143 was revised to emphasize precautions in shop practice in punching holes. The subcommittee recommended that the above three specifications be reapproved as standard with revisions made as outlined. Other subjects under consideration are inspection of galvanizing and regalvanizing specifications for hardware and tower steel.

Subcommittee XIV on Sheet Tests (C. P. Larrabee, chairman) inspected the atmospheric exposure tests of corrugated sheets at Altoona and State College, Pa., on April 17, 1958. The report of each inspection follows:

Altoona, Pa.—The one remaining sheet was found to be perforated; therefore the complete results of the test at this site will be published in the 1960 report.

State College, Pa.—The results of this inspection will be published in the 1960 report.

Subcommittee XV on Wire Tests (F. M. Reinhart, chairman) made the annual inspection of the wires at Bridgeport, Conn., State College, Pa., and Pittsburgh, Pa., in April 1958. The wires at the other sites were inspected by the university inspectors at each site during the autumn of 1958.

A twenty-year report and discussion on the wire tests is in preparation.

Subcommittee XVI on Hardware Tests (A. Mendizza, chairman) prepared the report which appears as an Appendix to this report.[5] No inspection was made in 1958 of the 1929 hardware specimens undergoing atmospheric exposure tests at State College, Pa. However an inspection was made on April 21, 1959, the results of which will appear in the 1960 report.

This report has been submitted to letter ballot of the committee which consists of 103 members; 68 members returned their ballots, of whom 61 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

Henry F. Hormann,
Chairman.

C. P. Larrabee,
Secretary.

---

[5] See p. 133.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-5 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specification for:

Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils, and Cut Lengths (A 93 - 58 T),
Long Terne Iron or Steel Sheets, Coils, and Cut Lengths (A 308 - 58 T), and
1.25 oz Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets (A 361 - 59 T).

These recommendations were accepted by the Standards Committee on October 27, 1959, and the revisions appear in the 1959 Supplement to Book of ASTM Standards, Part 1.

APPENDIX

PRELIMINARY DATA ON 1958 ATMOSPHERIC EXPOSURE OF HARDWARE SPECIMENS

REPORT OF SUBCOMMITTEE XVI ON HARDWARE TESTS, ASTM COMMITTEE A-5 ON CORROSION OF IRON AND STEEL

A test program designed to evaluate the weathering characteristics of metallic coatings on ferrous basis metals for possible use on hardware materials was initiated in 1952. This program was planned with the thought of supplementing the tests begun some 30 years ago and was designed primarily to furnish additional information, particularly with regard to the use of low-alloy high-strength steels.

COATINGS

The program that was ultimately agreed upon by the members of Subcommittee XVI comprises the following coatings:

Hot-dipped zinc (Code 5),
Hot-dipped aluminum (Code 6),
Hot-dipped aluminum (Code 7),
Electroplated zinc (Code 8),
Sprayed zinc (Code 9), and
Sprayed aluminum (Code 10).

Hot-dipped zinc coatings are by far the most widely used metallic finishes for outdoor applications and are therefore included as a standard for comparison. Hot-dipped aluminum coatings are receiving increasingly greater attention by hardware users. Since most hot-dipped aluminum coating processes are proprietary or licensed processes, it was decided to test coatings from two different sources. It is believed that these are fairly representative of current practices. Sprayed zinc and sprayed aluminum coatings have been included because of the popularity of these types of coatings for certain specific applications. Electroplated zinc was added as another yardstick of comparison both because of the large mass of data available about its performance outdoors and also because it is used, although to a rather limited extent, for some outdoor applications.

It was decided that insofar as possible these coatings should be applied in thicknesses ranging from 2 to 2.5 mils. It was felt that this would permit comparative evaluation of the various coatings within reasonable exposure time. In addition, uncoated specimens of the carbon steel and the four low-alloy steels in the form of flat panels were also exposed to the selected environments.

BASIS METALS

The following ferrous basis metals were chosen for this program:

Carbon steel (Code 1),
Low-alloy steel I (Code 2),
Low-alloy steel II (Code 3),
Low-alloy steel III (Code 4),
Low-alloy steel IV (no code number),
Nodular iron, as cast (Code 19),
Nodular iron, machined (Code 20), and
Malleable iron (Code 21).

Carbon steel is to serve as a standard of comparison. The low-alloy steels have been chosen because of their comparatively high strength and better corrosion

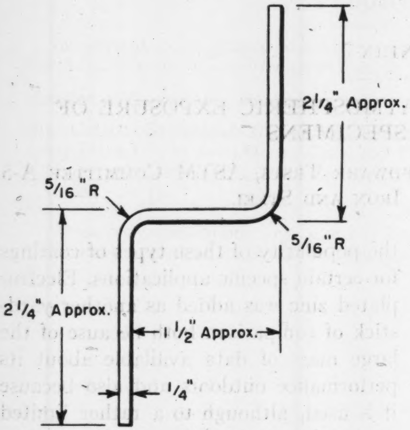


FIG. 1.—Bent Panel Hardware Test Specimen.

TABLE I.—COMPOSITION OF LOW-ALLOY STEEL SAMPLES.

Alloy	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Nickel	Chromium	Zirconium
	REPORTED COMPOSITION IN PER CENT								
I......	0.12 max	0.50 to 1.00	0.06 to 0.12	0.05 max	0.10 to 0.50	0.30 to 0.70	0.25 to 0.75	0.40 to 1.00	...
II......	0.08 to 0.15	0.50 to 0.75	0.04 max	0.05 max	0.60 to 0.90	...	...	0.50 to 0.65	0.05 to 0.15
III......	0.15 max	0.60 max	0.05 to 0.10	0.05 max	...	0.75 to 1.25	0.50 to 2.00	...	...
IV......	0.12 max	0.20 to 0.50	0.07 to 0.15	0.05 max	0.25 to 0.75	0.25 to 0.55	0.65 max	0.35 to 1.25	...
	DETERMINED COMPOSITION, PER CENT								
I......	0.10	...	...	...	...	0.50	0.62	0.47	...
II......	0.17	...	...	...	...	0.06	0.02	0.64	0.03
III......	0.14	...	...	...	...	0.37	0.54	...	...
IV......	0.10	...	...	...	...	0.24	0.30	1.10	...

resistance relative to carbon steel. Composition wise, they are characterized by small contents of copper, nickel, and chromium. Table I shows the nominal composition of these alloys. Nodular iron is essentially a cast iron in which the graphitic component is in the form of small spheres. It is highly ductile and heat treatable and is of some interest to hardware users. Malleable iron has been included because of the desirable physical and chemical properties peculiar to this material.

SPECIMEN FORM

In contrast to the earlier tests where the specimens used were in many cases actual hardware items, such as knockout boxes, nails, hinges, etc., the specimen form in the present test was limited to three basic shapes:

(*a*) 4 by 6 by ¼ in. flat panels,

(*b*) 4 by 6 by ¼ in. panels which had been cold bent prior to finishing in the form shown on Fig. 1, and

(*c*) Round rods ¾ in. in diameter and 6 in. long.

It was felt that these three basic forms would answer all major questions regarding the weathering characteristics of a finish as it might be affected by the geometry of a part. The bent panels especially were designed to reveal any abnormal effects on the performance of the coating due to the cold working of the basis metal. The panels and rods are identified as to basis metal, type of coating, and exposure location by appropriately drilled small holes and notches as shown in Fig. 2.

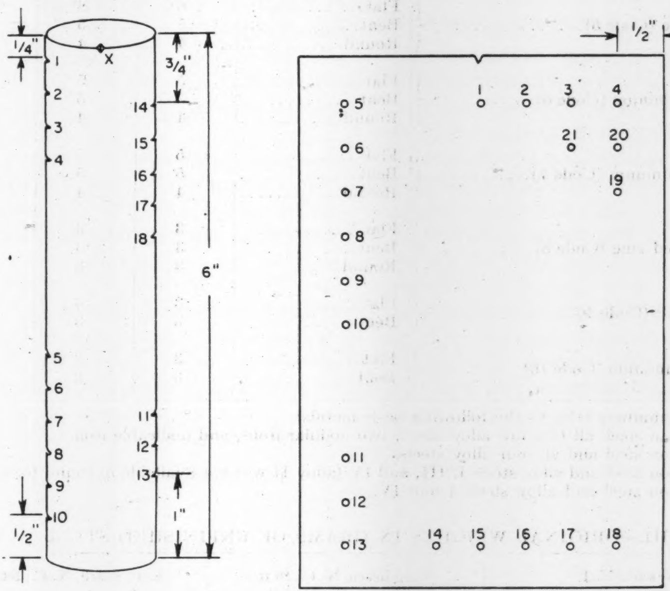


FIG. 2.—Identification of Specimens.
Distance Between ℄ of Notches 3/8 in.; of holes 1/2 in.
Explanation of Holes and Notches

1—Carbon steel.
2—Low-alloy steel I.
3—Low-alloy steel II.
4—Low-alloy steel III.
5—Hot-dipped zinc.
6—Hot-dipped aluminum 6.
7—Hot-dipped aluminum 7.
8—Electroplated zinc.
9—Sprayed zinc.
10—Sprayed aluminum.
11—New York area exposure.
12—800 ft. Kure Beach exposure.
13—80 ft. Kure Beach exposure.
14, 15, 16, 17, 18 } Panel numbers.
19—Nodular iron (as cast).
20—Nodular iron (machined).
21—Malleable iron.
No notch or no hole—low-alloy steel IV.
X—Locating notch.

TEST SITES

Three environments were chosen for these tests. The industrial environment of the New York Area (Newark, N. J.) test site (Code 11) and two marine environments at Kure Beach, N. C., located respectively 800 (Code 12) and 80 ft (Code 13) from the ocean. It was felt that these environments are representative of some of the most severe exposure conditions encountered within the continental United States. Exposure at a rural site was at first considered but ruled out because of an anticipated extremely long test duration in such a mild environment.

TABLE II.—EXPOSURE SCHEDULE.

Coating	Specimen Shape	Basis Steels Exposed at		
		Newark	Kure Beach 800-ft site	Kure Beach 80-ft site
Hot-dip zinc (Code 5)	Flat	8[a]	8[a]	8[a]
	Bent	5	5	5
	Round	4	4	4
Hot-dip aluminum (Code 6)	Flat	5	5	5
	Bent	5	5	5
	Round	4	4	4
Hot-dip aluminum (Code 7)	Flat	5	5	5
	Bent	5	5	5
	Round	4	4	4
Electroplated zinc (Code 8)	Flat	3	3	3
	Bent	3	3	3
	Round	3	3	3
Sprayed zinc (Code 9)	Flat	3	3	...
	Bent	3	3	...
Sprayed aluminum (Code 10)	Flat	3	3	...
	Bent	3	3	...

[a] These numbers refer to the following basis metals:
8—Carbon steel, all four low alloy steels, two nodular irons, and malleable iron.
5—Carbon steel and all four alloy steels.
4—Carbon steel and alloy steels I, III, and IV (alloy II was not available in round forms).
3—Carbon steel and alloy steels I and IV.

TABLE III.—ORIGINAL WEIGHTS IN GRAMS OF UNFINISHED STEEL PANELS.

Newark, N. J.		Kure Beach, N. C., 80 ft		Kure Beach, N. C., 800 ft	
Panel Number	g	Panel Number	g	Panel Number	g
0-5-11-15	776.13	0-5-13-15	775.00	0-5-12-15	768.20
0-5-11-16	767.56	0-5-13-16	763.20	0-5-12-16	758.05
0-5-11-17	771.32	0-5-12-18	764.65	0-5-12-17	750.00
1-5-12-14	805.38	1-5-13-15	826.95	1-5-12-17	808.55
1-5-12-15	812.26	1-5-13-16	831.45	1-5-12-18	799.85
1-5-12-16	816.10	1-5-13-17	824.25	2-5-12-14	750.95
2-5-11-15	750.00	2-5-13-16	769.85	2-5-12-16	754.50
2-5-11-17	754.28	2-5-13-17	763.45	2-5-12-17	765.10
2-5-11-18	757.38	3-5-12-14	738.25	3-5-12-16	736.20
3-5-12-15	741.38	3-5-13-17	735.25	3-5-12-18	733.05
3-5-11-17	737.92	4-5-13-14	742.35	4-5-12-14	756.20
3-5-11-18	754.00	4-5-13-16	769.70	4-5-12-15	749.15
4-5-11-15	765.82	21-13-14	703.20	4-5-12-16	746.25
4-5-11-17	758.45	21-13-16	687.30	21-12-14	669.45
4-5-11-18	756.15	21-13-17	718.20	21-12-15	690.55
21-11-17	717.34			21-12-17	736.00
21-12-16	690.21				
21-11-17	694.18				

EXPOSURE SCHEDULE

In order to keep the test program within reasonable limits, not all the coatings and steels investigated were exposed to all three test sites. The exposure schedule outlined in Table II shows the arrangement of the panels and rods at the test locations. It is believed that this will yield the maximum amount of information with the minimum number of panels.

Each coating - basis metal combination was exposed in quintuplicate. The four uncoated low-alloy steels, carbon steel, and malleable iron flat panels were exposed in triplicate (some only in duplicate) at the three test sites. A total of 830 coated and 49 uncoated panels and rods have been placed on exposure. For the purpose of obtaining eventual weight loss data, the uncoated steel panels were weighed prior to exposure. These weights are recorded in Table III.

COATING PROCEDURES

Hot-Dipped Zinc:

The panels and rods were processed following the conventional procedure of pickling, rinsing, fluxing, hot dipping, and centrifuging to remove excess metal. In order to effect a quick setting of the coating metal, the specimens were quenched in cold water immediately following the centrifuging operation.

The processor supplied the following remarks which may be pertinent in evaluating and appraising the performance of the hot-dipped zinc coatings:

1. In an attempt to produce coatings of the desired thickness, the time of immersion of most steels in the zinc bath (about 2 min) was shortened very considerably over that normally employed. The nodular iron panels had to be immersed about 6 to 7 min in order to achieve acceptable coverage. The resultant thickness was therefore expected to be considerably greater.
2. Rolled-in scale and badly rusted surfaces of some panels resulted in a finish coating with bare or black spots. Prolonged pickling to remove all imperfections had to be avoided in many instances so as not to damage the steel surface excessively.

Hot-Dipped Aluminum (Code 6):

The panels and rods were coated with aluminum using the following schedule:

1. Heated in furnace for 0.5 hour at 1000 F to remove surface grease,
2. Immersed 1 min in 10 per cent hydrochloric acid solution at room temperature to remove mill scale,
3. Rinsed in water and immersed in clean 15 per cent hydrochloric acid at room temperature for 5 min,
4. Rinsed in water and immersed in a "pre-dip" aqueous solution of alkali metal salts for 20 sec at 180 F, and
5. Dried and immersed in aluminizing bath at 1325 F for 1 min.

The nominal composition of the aluminizing bath is as follows: Silicon, 2.5 per cent; iron, 0.5 to 2.0 per cent; chromium, 0.2 per cent; magnesium, 0.1 per cent; titanium, 0.04 per cent.

Hot-Dipped Aluminum (Code 7):

The procedure in applying aluminum coatings by this patented process consisted essentially in degreasing the panels by chlorinated solvent or emulsion, acid pickling to remove heavy scale or rust, drying, preheating, fluxing, aluminizing at 1300 to 1320 F for 2 min, followed by a rinse in the fluxing bath to remove excessive aluminum. It is believed that this would result in an aluminum overlay thickness of 0.0005 to 0.001 in. and an aluminum-iron alloy layer about 0.002 in. thick.

Electroplated Zinc:

This finish was applied from a conventional cyanide bath. Comments regarding the cleaning of the samples for the hot-dipped zinc coatings apply also to the electroplated zinc panels.

Sprayed Zinc and Sprayed Aluminum:

The treatment of these panels was as follows:

1. Pressure blast at 60 psi with 20- to 40-mesh aluminum oxide abrasive to obtain surfaces suitable to receiving metal sprayed coatings.
2. Zinc and aluminum were sprayed on to a weight equivalent thickness of 0.002 to 0.0025 in. However, because of porosity, the as-sprayed coatings measured 0.003 to 0.0035 in.
3. The sprayed metal coatings were sealed with a thin sprayed coat of primer of the poly(vinyl butyral) type containing phosphoric acid and zinc chromate (wash primer) followed by a coat of aluminum vinyl. The addition of these sealing coats increased the thickness by approximately 0.001 in.

THICKNESS MEASUREMENTS

Hot-Dipped Zinc and Aluminum Flat and Bent Panels:

The following describes in detail the procedure followed by a task group in obtaining and analyzing the thickness measurements for four types of coatings on the flat and bent panels. The coatings measured and analyzed are: hot-dipped zinc, the two types of hot-dipped aluminum, and the electroplated zinc.

In deciding on the gage to use and the pattern and number of readings to be taken, two objectives were established:

1. Measurements should give average coating thickness on each panel, and
2. Some measure of the variability of the coating on a macroscale should be obtained.

From the average coating thickness on each panel, the variation in coating thickness between panels of the same type (same shape, base, and coating) and between different types can readily be calculated. This information is necessary to interpret intelligently the results of the exposure tests if time to failure of the coating is taken as the criterion.

Variations in coating thickness on a microscale should not have a marked effect on the life of anodic coatings such as zinc, as these coatings will provide cathodic protection over small discontinuities in the coating. Coating thickness variations on a larger scale will be more important as early rusting will occur at the larger thin areas. Aluminum coatings may, under certain conditions, also provide similar protection.

Coating Thickness Gage.—From previous experience it was decided that a thickness gage of the permanent magnetic type would be the most suitable for this test. This gage has good precision and reasonable accuracy when properly calibrated.

Pattern of Measurements.—A preliminary test was first made to determine the number and pattern of readings to be taken on each panel and to check the standardization of the gage. A statistical study of the thickness gage measurements showed that single measurements at about seven positions on the panels would give a good average coating thickness. The standard deviation of the readings would give a measure of the coating uniformity. Therefore it was decided to measure the panels as follows:

(*a*) *Flat Panels.*—Single measurements at nine positions on the two diagonals of the panel.

(*b*) *Bent Panels.*—Single measurements at eight positions, four on each of the parallel sections. It was not possible to take any measurements on the perpendicular section of the bent panels with the gage employed.

The positions at which the measurements were made are shown in Fig. 3.

Calibration of Gage.—The thickness gage was always calibrated using measured brass shims on a bare panel of the same steel as the specimens to be measured. Where necessary, the basis metal was demagnetized prior to using the gage.

Standardization.—The original intention was to use microscopic measurement of sections to standardize the magnetic thickness gage. It was found, however, that most of the coatings were too irregular to obtain an accurate check by this method. Stripping tests were therefore used as the standard for comparison. One panel of each combination of base and coatings was used for this test. Two rectangular panels approximately 2 by 2½ in. were taken from each panel, one from the top half and one from the bottom half. Ten gage measurements were made in each specimen area by each operator. The specimens were then cut out and the area was measured accurately. From this point the zinc-coated specimens and aluminum-coated specimens were treated differently as follows:

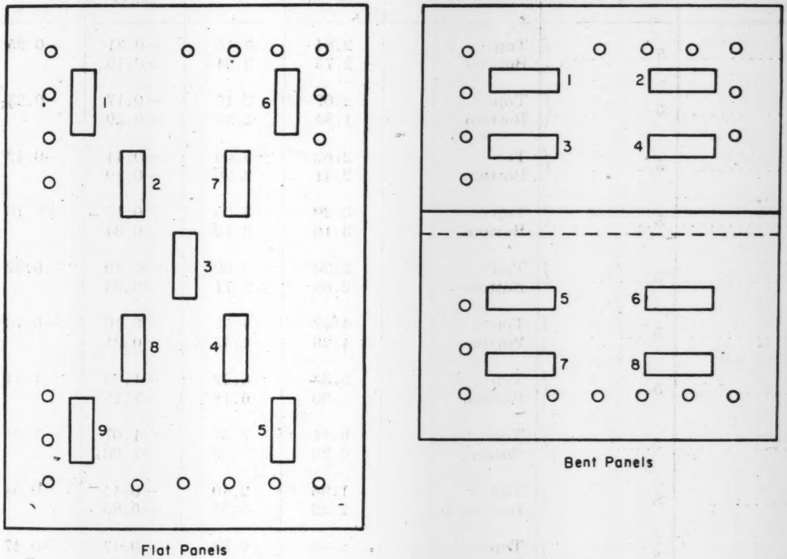


FIG. 3.—Template Positions for Coating Thickness Measurements.

1. *Zinc-Coated Specimens.*—The specimens were weighed and then the face not measured was coated with paraffin wax. The coating was stripped from the measured face with 50 per cent hydrochloric acid. The average coating thickness was calculated from the weight loss assuming a density of 7.14 g per cu cm for the coating. As the density of the iron-zinc alloy layers is very close to that of zinc, the error due to this assumption will be small. The results of this test are shown in Table IV. With one exception the measured thickness is greater than the thickness calculated from weight loss. A correction factor has been obtained by averaging the difference between measured and calculated thickness for the two specimens from each panel. This factor can be applied to the average measured thickness of specimens with

the same combination of base and coating.

2. *Aluminum-Coated Specimens.*—As the stripping solution for the aluminum coatings is used hot, paraffin could not be used, so the coating on the unmeasured face was removed by grinding. The specimens were then weighed and chemically stripped. After stripping the specimens were reweighed and the coating weight was calculated. The conversion of coating weight to average coating thickness is difficult for aluminum coatings. The density of aluminum is 2.77 g per cu cm and that of $FeAl_3$ is 3.84 g per cu cm. To calculate coating thickness accurately, the proportion of alloy layer in the coating must be known. Microsections showed this proportion varies greatly in each specimen. However a rough estimate of the percentage iron-aluminum alloy in each coating was made from the microsections taken from the same panels as the stripping specimens. Using these figures and the above densities the coating thicknesses were calculated. The results are shown in Table V. The thickness calculated from weight loss is within ± 15 per cent of the average thickness by measurement (average of 10 readings by each operator). These values are too approximate to use in calculating a correction factor as was done for the zinc coatings.

TABLE IV.—RESULTS OF STRIPPING TEST ON ZINC COATINGS.

Basis Metal	Coating	Specimen Face	Average Thickness Calculated from Weight Loss, mils	Average Thickness by Measurement, mils	Difference Between Calculated and Measured Values	Correction Factor
0	5	Top	2.84	3.15	−0.31	−0.25
		Bottom	2.75	2.94	−0.19	
1	5	Top	2.02	2.19	−0.17	−0.23
		Bottom	1.84	2.13	−0.29	
2	5	Top	2.62	3.06	−0.44	−0.42
		Bottom	2.41	2.80	−0.39	
3	5	Top	3.20	3.05	0.15	+0.10
		Bottom	3.16	3.12	0.04	
4	5	Top	2.20	2.60	−0.40	−0.52
		Bottom	2.08	2.71	−0.63	
19	5	Top	4.42	4.52	−0.10	−0.16
		Bottom	4.26	4.47	−0.21	
20	5	Top	5.33	6.42	−1.09	−1.14
		Bottom	5.00	6.18	−1.18	
21	5	Top	6.51	7.58	−1.07	−1.04
		Bottom	6.20	7.20	−1.00	
0	8	Top	1.96	2.40	−0.44	−0.64
		Bottom	2.22	3.07	−0.85	
1	8	Top	2.36	2.83	−0.47	−0.47
		Bottom	2.26	2.73	−0.47	
2	8	Top	1.80	2.40	−0.60	−0.68
		Bottom	2.01	2.76	−0.75	

Analysis of Results.—From the coating thickness measurements on each panel the following were calculated:

$\bar{X}$ = Average measured coating thickness for each panel, an average of the eight or nine measurements on the panels.

S = Standard deviation of measurements on each panel. This figure is a measure of the coating uniformity on that panel but also includes a factor of measurement error.

$\bar{\bar{X}}$ = Average coating thickness for group of 15 panels of the same shape, base, and coating.

$S_{\bar{x}}$ = Standard deviation of the $\bar{X}$'s of panels in the group. This figure is a measure of variation of coating thickness between panels in the group.

S' = Average (geometric) of standard deviations for panels in the group. This figure is a measure of coating variation within panels for the whole group. It does not include any factor of the variation between panels. This figure may be used to compute the minimum significant difference in average coating thickness between two panels in the same group. That is, if $\bar{X}$ for one panel is more than S' different from $\bar{X}$ for another panel in the same group, there is a good chance that the coating thicknesses of the two panels are significantly different.

The results for groups are summarized in Table VI. The correction factors calculated from the results of the stripping tests on the zinc-coated specimens are also tabulated along with a corrected average coating thickness which is $\bar{X}$ with the above correction factor applied.

TABLE V.—RESULTS OF STRIPPING TESTS ON ALUMINUM COATINGS.

Basis Metal	Coating	Specimen Face	Average Thickness Calculated from Weight Loss, mils	Average Thickness by Measurement, mils	Difference, Between Calculated and Measured Values, mils
0	6	Top	2.21	2.20	0.01
		Bottom	2.23	2.26	−0.03
1	6	Top	1.99	1.92	0.07
		Bottom	2.07	2.17	−0.10
2	6	Top	1.78	1.98	−0.20
		Bottom	2.29	2.57	−0.38
3	6	Top	1.95	2.15	0.20
		Bottom	2.69	2.75	−0.06
4	6	Top	2.40	2.37	0.03
		Bottom	3.19	3.27	−0.08
0	7	Top	3.67	3.91	−0.24
		Bottom	3.80	4.17	−0.37
1	7	Top	3.61	3.59	0.02
		Bottom	3.69	3.55	0.14
2	7	Top	5.26	5.04	0.22
		Bottom	5.04	5.01	0.03
3	7	Top	3.74	4.05	−0.31
		Bottom	3.20	3.45	−0.25
4	7	Top	5.53[a]	4.20	...
		Bottom	4.56	4.13	−0.43

[a] Some coating not ground off opposite face, therefore weight loss too high.

TABLE VI.—SUMMARY OF COATING THICKNESS MEASUREMENTS.

All readings in mils (0.001 in.)

FLAT PANELS

	Zinc Coatings										
	Hot-Dip (Code 5)								Electroplated (Code 8)		
Basis Metal	0	1	2	3	4	19	20	21	0	1	2
$\overline{\overline{X}}$	3.14	2.25	3.26	2.83	3.22	3.92	6.17	7.18	2.59	2.55	3.15
$S_{\bar{x}}$	0.21	0.20	0.29	0.34	0.60	0.51	0.33	0.26	0.39	0.45	0.35
S'	0.19	0.26	0.34	0.39	0.38	0.30	0.29	0.42	0.21	0.30	0.32
Correction factor	−0.25	−0.23	−0.42	+0.10	−0.52	−0.16	−1.14	−1.04	−0.64	−0.47	−0.68
Corrected average	2.89	2.02	2.84	2.93	2.70	3.76	5.03	6.14	1.95	2.08	2.47

	Aluminum Coatings									
	Hot-Dip (Code 6)					Hot-Dip (Code 7)				
Basis Metal	0	1	2	3	4	0	1	2	3	4
$\overline{\overline{X}}$	2.27	2.34	2.27	2.43	2.84	3.52	3.23	3.55	3.55	3.22
$S_{\bar{x}}$	0.26	0.31	0.33	0.33	0.43	0.69	0.74	0.45	0.89	0.58
S'	0.43	0.30	0.41	0.37	0.38	0.26	0.24	0.26	0.28	0.20

BENT PANELS

	Zinc Coatings							
	Hot-Dip (Code 5)					Electroplated (Code 8)		
Basis Metal	0	1	2	3	4	0	1	2
$\overline{\overline{X}}$	2.13	2.14	2.53	2.40	2.74	2.60	2.21	2.93
$S_{\bar{x}}$	0.11	0.23	0.18	0.15	0.42	0.46	0.42	0.50
S'	0.22	0.27	0.29	0.29	0.52	0.49	0.49	0.53
Correction factor	−0.25	−0.23	−0.42	+0.10	−0.52	−0.64	−0.47	−0.68
Corrected average	1.88	1.91	2.11	2.50	2.22	1.96	1.74	2.25

	Aluminum Coatings									
	Hot-Dip (Code 6)					Hot-Dip (Code 7)				
Basis Metal	0	1	2	3	4	0	1	2	3	4
$\overline{\overline{X}}$	1.76	2.30	1.86	1.61	2.28	2.21	2.53	2.52	2.38	2.55
$S_{\bar{x}}$	0.11	0.23	0.11	0.14	0.23	0.34	0.23	0.39	0.34	0.19
S'	0.35	0.38	0.32	0.38	0.38	0.20	0.22	0.28	0.18	0.17

As explained under "Standardization," a correction factor for the aluminum-coated panels could not be calculated.

Complete results for every flat and bent panel are shown in Tables VII and VIII. These include $\bar{X}$ and S for each specimen, and $\bar{X}$, $S_{\bar{x}}$ and S' for each group of panels.

Sprayed Coatings:

The sprayed coatings received a weight equivalent coating of 0.002 to 0.0025 in. Because of the porous nature of the coating, the as-measured thicknesses ranged between 0.003 and 0.0035 in. As a result of sealing these coatings with vinyl enamels the ultimate thickness was of the order of 0.003 to 0.004 in.

Hot-Dip Zinc and Aluminum Round Rods:

The thicknesses of the coatings on the round rods were measured by another task group. All rods were measured with a magnetic gage. The thickness on a representative number of aluminum coated rods was measured by the stripping technique. Table IX shows the magnetic gage readings for all the round rods. Sections A, B, and C are areas where the measurements were taken and are located approximately 1, 3, and 5 in. from one end. Areas B and C are displaced radially by 120 and 240 deg respectively from area A.

In Table X are summarized the average, minimum, and maximum thicknesses of the two aluminum coatings on carbon steel and on three alloy steels obtained by direct measurements of round rods with a magnetic gage and by the stripping method. As there were no extra zinc-coated round rods available, thickness determination by the stripping method could not be obtained for these samples.

From the values shown in Table IX it can be seen that:

1. The thickness of coating on any one rod as determined by the magnetic gage is in most cases not very uniform from one end of the rod to the other, regardless of coating or basis steel.
2. The averages of each rod are for the most part significantly different within a set of five replicates.
3. More than 50 per cent of the thickness values for the hot-dip zinc coatings are in the range between 4 and 5.5 mils. The minimum thickness is 3 and the maximum 6.17 mils.
4. The majority of the aluminum coating Code 6 range between 3 and 4 mils, with a minimum of 2.16 and a maximum of 5.12 mils.
5. The thickness range for most of the aluminum Code 7 coatings lies between 2.5 and 4 mils, with a low of 1.02 and a high of 6.13 mils.
6. Electroplated zinc ranges mostly between 2 and 3 mils, having a minimum of 1.64 and a maximum of 3.76 mils.

From an inspection of the values in Table X, it is evident that the measurements of the two aluminum coatings on the round rods by the magnetic and by the stripping method are not directly comparable.

A comparison between Tables IX and Tables VII and VIII shows that the values of the coating thickness on the round rods are in general greater than those obtained on the flat and on the bent panels. Also, the uniformity of coating on the latter panels is of a higher order than on the rounds. This could be ascribed to different processing techniques used for applying the hot-dip coatings and to the highly uneven surface conditions and extremely wide variations in the thickness of the alloy layer, particularly as regards the aluminum coatings. The electroplated zinc coatings appear to be of about the same order of magnitude on all rod and panel forms.

TABLE VII.—RESULTS OF COATING THICKNESS MEASUREMENTS ON FLAT PANELS, MILS.

Basis Metal	Coating		New York Area Site					Kure Beach, N. C. 800-ft Site					Kure Beach, N. C. 80-ft Site					$\bar{\bar{X}}$	$S_{\bar{x}}$	S'
			14	15	16	17	18	14	15	16	17	18	14	15	16	17	18			
0...	5...	$\bar{X}$	3.29	3.42	2.92	3.23	3.01	2.78	2.96	3.47	3.13	2.89	3.18	3.08	3.43	3.18	3.10	3.14	0.21	....
		S	0.38	0.22	0.10	0.15	0.11	0.14	0.18	0.38	0.15	0.08	0.16	0.17	0.09	0.15	0.15	......	......	0.19
1...	5...	$\bar{X}$	2.44	2.24	2.16	1.86	2.70	2.24	2.37	2.18	2.06	2.01	2.32	2.26	2.21	2.29	2.40	2.25	0.20	....
		S	0.31	0.25	0.12	0.17	0.34	0.16	0.63	0.11	0.07	0.32	0.12	0.28	0.19	0.14	0.19	....	....	0.26
2...	5...	$\bar{X}$	3.66	2.81	3.14	3.08	3.74	3.66	3.01	3.20	3.36	3.09	3.59	3.11	3.04	3.03	3.31	3.26	0.29	....
		S	0.70	0.08	0.15	0.65	0.35	0.31	0.29	0.13	0.16	0.27	0.41	0.08	0.11	0.33	0.31	....	....	0.34
3...	5...	$\bar{X}$	2.63	2.54	2.86	3.18	2.72	2.98	2.87	3.77	2.47	2.38	2.62	2.71	2.72	2.87	3.08	2.83	0.34	....
		S	0.16	0.16	0.15	0.13	0.18	0.29	0.22	1.28	0.24	0.37	0.23	0.14	0.12	0.18	0.30	....	....	0.39
4...	5...	$\bar{X}$	4.11	3.31	4.19	2.88	2.30	3.43	2.51	2.74	2.69	2.53	3.90	3.49	3.62	3.07	3.51	3.22	0.60	....
		S	0.43	0.52	0.25	0.52	0.10	0.32	0.24	0.25	0.29	0.27	0.24	0.41	0.63	0.37	0.49	....	....	0.38
19...	5...	$\bar{X}$	4.77	2.47	4.88	4.81	4.36	4.38	2.83	4.62	....	4.87	4.28	4.51	2.77	4.36	4.83	3.92	0.51	....
		S	0.24	0.20	0.45	0.25	0.26	0.24	0.34	0.47	....	0.34	0.21	0.41	0.24	0.29	0.15	....	....	0.30
20...	5...	$\bar{X}$	6.47	6.38	5.97	6.29	6.57	....	6.22	6.03	6.03	6.04	5.51	6.14	5.63	6.48	6.57	6.17	0.33	....
		S	0.23	0.17	0.10	0.35	0.23	....	0.29	0.20	0.18	0.39	0.46	0.42	0.21	0.25	0.30	....	....	0.29
21...	5...	$\bar{X}$	7.23	7.06	7.36	6.89	7.43	7.66	7.10	7.42	6.99	6.94	6.77	6.92	7.39	7.03	7.44	7.18	0.26	....
		S	0.66	0.23	0.63	0.36	0.24	0.37	0.30	0.34	0.18	0.38	0.35	0.41	0.43	0.47	0.43	....	....	0.42
0...	6...	$\bar{X}$	1.77	2.30	2.37	2.39	2.51	2.01	2.32	2.31	2.10	2.44	2.21	2.77	1.91	2.08	2.58	2.27	0.26	....
		S	0.25	0.32	0.29	0.50	0.28	0.30	0.37	0.31	0.17	0.26	0.29	0.37	0.16	0.44	1.15	....	....	0.43
1...	6...	$\bar{X}$	2.27	1.86	2.48	2.22	2.03	2.47	2.63	2.28	3.17	2.48	2.18	2.47	2.33	1.99	2.26	2.34	0.31	....
		S	0.23	0.23	0.22	0.23	0.23	0.43	0.41	0.21	0.42	0.35	0.11	0.24	0.17	0.24	0.45	....	....	0.30
2...	6...	$\bar{X}$	2.23	1.90	1.91	2.06	2.09	2.16	2.06	2.16	2.47	2.74	2.70	2.19	2.98	1.96	2.39	2.27	0.33	....
		S	0.25	0.19	0.35	0.28	0.29	0.36	0.27	0.31	0.68	0.36	0.52	0.26	0.75	0.34	0.46	....	....	0.41
3...	6...	$\bar{X}$	2.40	1.87	2.30	2.32	2.57	2.46	2.17	2.73	2.43	2.43	3.31	2.14	2.33	2.22	2.70	2.43	0.33	....
		S	0.42	0.31	0.27	0.28	0.38	0.57	0.18	0.24	0.43	0.40	0.60	0.35	0.36	0.21	0.24	....	....	0.37

4. . .	6. . .	$\bar{X}$	2.66	2.92	4.06	3.32	3.14	2.38	2.69	2.76	2.42	2.79	2.66	2.71	3.02	2.77	2.43	2.84	0.43	. . . .
		S	0.37	0.31	0.80	0.28	0.51	0.24	0.24	0.26	0.34	0.21	0.35	0.29	0.35	0.47	0.26	. . . .	. . . .	0.38
0. . .	7. . .	$\bar{X}$	3.36	3.52	4.59	2.88	2.88	2.40	3.06	5.03	3.78	4.30	3.53	3.33	3.58	3.43	3.12	3.52	0.69	. . . .
		S	0.21	0.13	0.25	0.17	0.26	0.11	0.28	0.41	0.24	0.18	0.51	0.20	0.25	0.13	0.19	. . . .	. . . .	0.26
1. . .	7. . .	$\bar{X}$	2.51	3.70	2.64	2.19	2.72	3.29	3.79	3.88	4.07	3.80	2.28	2.64	4.70	3.19	3.01	3.23	0.74	. . . .
		S	0.09	0.29	0.14	0.16	0.22	0.29	0.23	0.33	0.25	0.24	0.09	0.18	0.43	0.28	0.09	. . . .	. . . .	0.24
2. . .	7. . .	$\bar{X}$	4.28	2.77	3.21	4.06	3.12	3.33	3.17	3.79	3.68	3.88	3.01	3.54	4.17	3.70	3.52	3.55	0.45	. . . .
		S	0.32	0.12	0.11	0.42	0.20	0.13	0.18	0.21	0.12	0.16	0.28	0.25	0.46	0.16	0.36	. . . .	. . . .	0.26
3. . .	7. . .	$\bar{X}$	2.74	4.60	2.76	2.12	3.07	4.68	4.71	2.87	3.59	3.14	4.54	3.60	3.21	4.81	2.84	3.55	0.89	. . . .
		S	0.36	0.28	0.30	0.19	0.21	0.34	0.31	0.19	0.15	0.33	0.35	0.32	0.15	0.35	0.14	. . . .	. . . .	0.28
4. . .	7. . .	$\bar{X}$	2.84	2.76	2.49	3.29	2.52	2.94	2.78	4.12	3.99	4.24	3.01	3.61	2.69	3.58	3.48	3.22	0.58	. . . .
		S	0.19	0.09	0.32	0.24	0.17	0.11	0.21	0.24	0.25	0.27	0.19	0.18	0.18	0.09	0.12	. . . .	. . . .	0.20
0. . .	8. . .	$\bar{X}$	2.58	2.54	2.84	2.69	2.81	2.70	2.56	1.91	2.48	2.87	3.56	1.91	2.51	2.53	2.41	2.59	0.39	. . . .
		S	0.21	0.22	0.22	0.14	0.27	0.18	0.35	0.16	0.18	0.19	0.16	0.18	0.22	0.22	0.13	. . . .	. . . .	0.21
1. . .	8. . .	$\bar{X}$	2.71	2.99	2.32	3.20	2.60[a]	2.96	3.06	3.13	2.20	2.14	2.09	2.17	1.68	2.50	2.53	2.55	0.45	. . . .
		S	0.26	0.28	0.29	0.34	0.33	0.42	0.52	0.43	0.25	0.27	0.18	0.14	0.19	0.11	0.27	. . . .	. . . .	0.30
2. . .	8. . .	$\bar{X}$	2.88	3.53	3.09	3.31	3.34[a]	3.12	3.04	2.89	2.61	3.22	4.14	3.03	3.06	3.17	2.89	3.15	0.35	. . . .
		S	0.20	0.24	0.39	0.21	0.48	0.29	0.32	0.35	0.29	0.26	0.40	0.40	0.29	0.25	0.34	. . . .	. . . .	0.32

[a] Front faces on these panels very poor, so back faces were also measured. Results for back faces were:

Specimen	1-8-11-18	2-8-11-18
$\bar{X}$	2.77	3.22
S	0.24	0.23

TABLE VIII.—RESULTS OF COATING THICKNESS MEASUREMENTS ON BENT PANELS, MILS.

Basis Metal	Coating		New York Area Site					Kure Beach, N. C. 800-ft Site					Kure Beach, N. C. 80-ft Site					$\bar{\bar{X}}$	$S_{\bar{x}}$	S'
			14	15	16	17	18	14	15	16	17	18	14	15	16	17	18			
0....	5...	$\bar{X}$	2.01	2.01	2.18	2.35	2.28	2.21	2.04	2.01	2.02	2.10	2.12	2.15	2.21	2.19	2.09	2.13	0.11	....
		S	0.20	0.34	0.16	0.22	0.30	0.20	0.19	0.22	0.12	0.14	0.26	0.27	0.19	0.11	0.22	....	....	0.22
1....	5...	$\bar{X}$	2.28	2.12	2.15	1.91	1.96	2.82	2.05	2.05	1.98	2.08	2.15	2.24	2.42	1.98	1.98	2.14	0.23	....
		S	0.32	0.27	0.27	0.25	0.23	0.45	0.16	0.23	0.14	0.26	0.35	0.29	0.33	0.24	0.13	....	....	0.27
2....	5...	$\bar{X}$	2.34	2.46	2.29	2.60	2.28	2.54	2.74	2.49	2.64	2.51	2.60	2.80	2.59	2.85	2.28	2.53	0.18	....
		S	0.33	0.38	0.19	0.24	0.27	0.32	0.23	0.30	0.39	0.29	0.26	0.35	0.26	0.15	0.24	....	....	0.29
3....	5...	$\bar{X}$	2.42	2.41	2.72	2.50	2.49	2.24	2.29	2.19	2.41	2.45	2.49	2.39	2.33	2.56	2.14	2.40	0.15	....
		S	0.44	0.14	0.28	0.27	0.14	0.15	0.26	0.08	0.22	0.37	0.43	0.22	0.18	0.51	0.18	....	....	0.29
4....	5...	$\bar{X}$	2.29	2.09	2.22	2.20	2.84	3.32	2.68	3.10	2.76	3.10	3.12	2.52	3.44	2.58	2.81	2.74	0.42	....
		S	0.15	0.22	0.21	0.16	0.53	0.67	0.27	0.40	1.33	0.56	0.50	0.59	0.26	0.33	0.22	....	....	0.52
0....	6...	$\bar{X}$	1.65	1.61	1.65	1.72	1.75	1.98	1.85	1.72	1.79	1.86	1.68	1.90	1.81	1.81	1.59	1.76	0.11	....
		S	0.30	0.18	0.12	0.20	0.21	0.38	0.51	0.41	0.31	0.40	0.31	0.45	0.48	0.48	0.22	....	....	0.35
1....	6...	$\bar{X}$	1.95	2.34	2.10	2.22	2.58	2.48	2.55	2.35	2.06	2.28	2.40	2.42	2.44	1.81	2.51	2.30	0.23	....
		S	0.05	0.33	0.45	0.58	0.26	0.38	0.34	0.31	0.19	0.31	0.49	0.72	0.27	0.15	0.32	....	....	0.38
2....	6...	$\bar{X}$	1.90	2.06	1.86	2.06	1.80	1.69	1.86	1.94	1.80	1.99	1.74	1.80	1.74	1.91	1.81	1.86	0.11	....
		S	0.38	0.35	0.30	0.47	0.34	0.18	0.27	0.31	0.12	0.44	0.37	0.31	0.28	0.28	0.31	....	....	0.32
3....	6...	$\bar{X}$	1.76	1.52	1.72	1.62	1.59	1.51	1.42	1.96	1.54	1.45	1.55	1.61	1.69	1.61	1.54	1.61	0.14	....
		S	0.39	0.23	0.36	0.45	0.39	0.35	0.33	0.67	0.41	0.21	0.26	0.40	0.30	0.42	0.32	....	....	0.38
4....	6...	$\bar{X}$	2.39	2.14	2.48	1.91	2.52	2.22	2.20	2.61	1.89	2.21	2.62	2.34	2.29	1.96	2.36	2.28	0.23	....
		S	0.20	0.21	0.61	0.22	0.32	0.33	0.13	0.39	0.20	0.34	0.29	0.40	0.64	0.33	0.61	....	....	0.38
0....	7...	$\bar{X}$	2.51	2.61	2.20	2.70	2.09	2.10	2.45	2.02	2.76	2.32	1.69	1.84	1.91	2.04	1.86	2.21	0.34	....
		S	0.23	0.11	0.33	0.13	0.20	0.15	0.21	0.21	0.33	0.17	0.08	0.11	0.24	0.14	0.18	....	....	0.20
1....	7...	$\bar{X}$	2.96	2.69	2.49	2.76	2.56	2.49	2.34	2.86	2.60	2.45	2.29	2.12	2.51	2.21	2.66	2.53	0.23	....
		S	0.23	0.30	0.21	0.17	0.14	0.07	0.20	0.18	0.24	0.29	0.11	0.14	0.31	0.22	0.26	....	....	0.22

2....	7...	$\bar{X}$	2.16	2.16	2.22	2.29	2.49	2.78	2.28	2.96	2.52	2.60	2.21	3.30	2.30	3.31	2.25	2.52	0.39	....
		S	0.21	0.23	0.12	0.25	0.19	0.35	0.18	0.37	0.38	0.40	0.22	0.44	0.09	0.36	0.15	....	....	0.28
3....	7...	$\bar{X}$	2.29	2.45	2.65	2.22	2.30	2.49	2.45	2.06	1.90	3.41	2.26	2.35	2.36	2.40	2.16	2.38	0.34	....
		S	0.11	0.11	0.15	0.18	0.17	0.11	0.20	0.20	0.16	0.20	0.12	0.19	0.24	0.13	0.28	....	....	0.18
4....	7...	$\bar{X}$	2.40	2.21	2.51	2.42	2.32	2.59	2.80	2.39	2.46	2.76	2.54	2.80	2.59	2.81	2.69	2.55	0.19	....
		S	0.31	0.11	0.23	0.19	0.13	0.15	0.16	0.10	0.12	0.21	0.15	0.14	0.16	0.22	0.08	....	....	0.17
0....	8...	$\bar{X}$	2.39	2.70	2.18	2.93	1.81	2.08	3.14	2.96	2.36	1.92	3.01	3.05	2.78	2.55	3.19	2.60	0.46	....
		S	0.61	0.50	0.49	0.45	0.38	0.41	0.47	0.45	0.40	0.45	0.61	0.47	0.54	0.67	0.37	....	....	0.49
1....	8...	$\bar{X}$	1.74	1.80	2.46	2.16	1.62	2.21	2.75	2.06	1.86	3.04	2.66	2.41	2.38	2.25	1.72	2.21	0.42	....
		S	0.49	0.61	0.50	0.47	0.37	0.46	0.53	0.37	0.42	0.47	0.50	0.64	0.52	0.55	0.31	....	....	0.49
2....	8...	$\bar{X}$	2.52	3.39	2.98	3.36	2.35	3.69	3.36	2.81	2.61	3.45	2.50	3.12	2.99	1.85	3.02	2.93	0.50	....
		S	0.46	0.51	0.50	0.46	0.71	0.40	0.33	0.58	0.72	0.53	0.32	0.47	0.44	0.48	0.81	....	....	0.53

TABLE IX.—RESULTS OF COATING THICKNESS MEASUREMENTS ON ROUND RODS, MILS.

Basis Metal Coating Test Site	Rod No. 14			Rod No. 15			Rod No. 16			Rod No. 17			Rod No. 18			Average				
	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C	14	15	16	17	18
0-5-11	5.20	3.85	3.95	5.50	3.82	4.78	5.42	6.58	6.85	6.17	4.38	4.55	3.85	3.85	4.45	4.33	4.70	6.28	5.03	4.05
0-5-12	3.50	4.02	5.03	5.60	5.32	5.40	5.40	3.76	5.40	4.95	5.40	5.50	5.80	5.40	4.70	4.17	5.44	4.89	5.28	5.30
0-5-13	4.85	4.38	4.95	5.50	5.32	5.12	4.23	4.95	6.30	6.17	4.10	5.32	6.43	5.40	5.20	4.73	5.31	5.16	5.19	5.68
1-5-11	7.25	4.02	6.43	4.87	5.32	6.43	4.95	2.61	5.40	4.70	4.70	5.20	6.58	4.70	2.48	5.90	5.54	4.32	4.87	4.59
1-5-12	4.02	4.02	4.23	4.70	4.95	5.40	3.55	2.79	5.03	2.83	4.87	3.40	7.10	2.79	2.90	4.09	5.02	3.12	3.70	3.26
1-5-13	4.95	4.38	5.89	5.20	3.24	3.13	3.63	5.03	5.32	5.69	4.95	6.03	5.69	5.32	2.04	5.07	3.86	4.66	5.56	4.35
2-5-11	5.50	5.80	4.32	5.32	4.70	3.85	6.58	4.70	5.60	4.10	4.70	3.82	5.12	3.95	5.12	5.21	4.62	5.53	4.21	4.73
2-5-12	5.80	6.70	6.17	4.63	5.20	6.30	6.43	2.90	2.79	4.02	3.29	...	6.30	5.50	7.25	6.22	5.38	4.04	3.66	6.35
2-5-13	3.13	5.32	4.45	5.60	5.40	...	3.50	3.70	4.23	4.95	5.60	6.95	4.78	2.83	4.38	4.30	5.50	3.81	5.83	4.00
4-5-11	4.16	7.08	6.43	5.12	5.32	7.55	5.40	3.20	3.55	6.58	4.16	5.60	5.40	5.20	4.78	5.89	6.00	4.05	5.45	5.13
4-5-12	3.05	4.32	3.20	4.16	5.60	2.59	4.23	2.75	2.90	5.32	5.40	4.32	4.70	5.20	4.95	3.52	4.12	3.19	5.01	4.95
4-5-13	4.87	4.87	4.38	5.69	2.36	5.12	3.24	3.40	5.03	3.10	2.79	3.24	3.85	4.63	5.03	4.71	4.39	3.89	3.04	4.50
0-6-11	7.10	4.10	3.24	3.10	3.24	3.14	1.55	2.65	2.83	3.40	2.79	3.76	2.95	3.20	2.95	4.61	3.16	2.34	3.32	3.03
0-6-12	3.82	3.24	3.00	2.90	3.34	3.95	3.40	1.72	2.95	2.95	2.93	3.00	8.00+	3.85	3.20	3.35	3.39	2.69	2.96	5.00+
0-6-13	5.32	3.29	5.12	3.55	3.55	2.86	2.72	4.70	2.52	3.82	3.46	2.95	3.76	3.24	2.83	4.58	3.32	3.31	3.41	3.28
1-6-11	2.25	2.54	1.96	5.32	3.76	5.20	2.72	2.48	5.03	3.70	5.12	3.05	3.24	2.95	2.25	2.25	4.76	3.74	3.99	2.81
1-6-12	3.50	6.58	6.43	3.55	4.10	3.76	2.30	1.81	2.42	6.85	3.95	2.28	3.29	4.23	2.45	5.50	3.80	2.18	4.36	3.32
1-6-13	3.00	2.58	2.54	3.40	3.40	3.70	3.40	3.50	3.85	3.34	1.74	3.50	4.10	2.20	2.30	2.71	3.50	3.58	2.86	2.87
2-6-11	3.14	3.10	3.50	3.10	3.14	3.85	4.10	3.85	3.55	4.78	3.50	2.90	2.48	2.45	3.00	3.27	3.36	3.73	3.73	2.73
2-6-12	3.24	3.70	2.95	2.90	3.34	3.76	3.95	2.93	3.50	3.24	3.85	4.38	5.12	2.45	2.08	3.29	3.33	3.46	3.86	3.22
2-6-13	2.93	3.85	3.55	1.81	4.02	4.10	3.50	3.34	3.29	2.79	2.69	3.40	2.48	3.70	1.70	3.44	3.31	3.37	2.96	2.63
4-6-11	3.05	3.40	3.40	4.16	4.16	3.55	3.63	3.63	3.82	3.10	3.50	4.45	5.89	4.02	3.95	3.28	3.96	3.69	3.68	4.62
4-6-12	3.40	3.85	4.23	4.23	4.32	5.40	4.02	2.75	2.93	4.87	4.16	3.24	2.36	2.54	2.42	3.82	4.65	3.23	4.09	2.44
4-6-13	3.34	3.34	...	6.70	2.52	3.29	4.23	4.63	3.63	3.29	3.46	4.16	3.50	3.70	3.55	3.34	4.17	4.16	3.64	3.58
0-7-11	2.79	3.50	2.61	5.40	6.03	5.40	2.58	2.30	1.58	2.95	2.93	2.52	4.23	5.03	4.32	2.93	5.61	2.15	2.87	4.53
0-7-12	1.20	1.31	0.68	4.38	4.55	3.55	3.50	7.55	4.55	6.58	5.60	7.25	1.63	4.16	3.63	1.06	4.16	5.20	4.68	3.14
0-7-13	3.85	4.32	4.23	7.10	3.40	4.23	5.89	5.12	4.16	5.20	4.38	7.25	3.95	3.24	5.03	4.13	4.91	5.06	5.61	4.07
1-7-11	3.46	2.83	4.16	3.95	2.86	2.52	3.14	3.29	3.34	3.29	2.42	3.24	3.95	4.45	2.93	3.48	3.11	3.26	2.98	3.78
1-7-12	3.34	3.00	3.55	4.63	2.72	3.82	3.40	3.29	3.46	3.40	2.48	3.76	3.20	3.63	4.55	3.33	3.72	3.38	3.21	3.79
1-7-13	4.70	3.29	4.10	2.90	4.87	3.34	3.29	6.43	4.95	3.34	4.32	3.70	3.00	4.70	4.32	4.03	3.70	4.89	3.79	4.01
2-7-11	7.40	7.70	3.34	2.58	2.20	2.42	5.12	7.40	4.55	5.69	5.89	3.82	3.46	2.93	3.34	6.15	2.40	5.69	5.13	3.24
2-7-12	4.23	3.34	2.83	6.58	4.78	4.63	4.55	2.86	3.00	4.16	3.20	2.95	2.95	2.69	2.52	3.47	5.33	3.43	3.44	2.73
2-7-13	6.70	3.82	3.82	5.89	2.86	3.40	4.70	3.24	6.17	2.28	3.14	2.58	3.82	2.79	3.34	4.78	4.05	4.70	2.67	3.32
4-7-11	4.10	4.55	3.29	4.38	5.12	2.90	4.02	3.24	3.46	5.03	4.10	4.38	2.90	3.05	3.05	3.98	4.13	3.91	4.50	3.00
4-7-12	3.40	3.63	4.70	2.72	3.70	2.93	6.85	3.76	3.95	3.85	3.29	2.86	3.55	3.20	3.20	3.91	3.12	4.85	3.33	3.32
4-7-13	4.32	3.76	3.24	4.63	3.85	3.40	4.16	3.00	4.23	3.63	4.95	4.63	4.63	3.34	4.16	3.77	3.96	3.79	4.40	4.04
0-8-11	1.83	2.61	2.25	3.29	2.90	2.45	2.90	2.33	3.76	1.75	2.65	3.05	2.61	3.34	2.54	2.23	2.88	2.99	2.48	2.83
0-8-12	...	...	...	2.69	1.83	2.42	2.83	1.79	2.90	...	...	...	1.93	2.65	3.20	...	2.31	2.51	...	2.59
0-8-13	2.66	3.24	3.29	2.86	2.28	1.96	2.86	1.89	2.42	3.63	2.38	2.86	2.72	3.14	3.14	3.06	2.37	2.39	2.96	3.00
1-8-11	2.48	2.93	1.87	3.50	2.58	1.68	1.83	1.50	1.60	2.75	1.66	2.30	2.86	2.38	2.20	2.76	2.59	1.64	2.23	2.45
1-8-12	2.10	1.70	2.52	1.72	2.58	3.05	1.85	2.69	3.24	1.74	2.75	1.91	2.54	2.10	2.48	2.11	2.45	2.59	2.13	2.37
1-8-13	2.10	1.87	3.95	3.40	1.77	3.50	1.83	1.85	3.46	3.46	3.63	2.36	3.40	2.48	2.13	2.64	2.89	2.38	3.15	2.67
2-8-11	3.20	2.86	2.06	2.93	1.35	2.58	2.22	2.93	3.09	1.85	2.52	3.46	3.46	3.76	2.83	2.71	2.29	2.75	2.61	3.35
2-8-12	2.25	2.52	0.92	2.20	1.55	3.00	3.63	2.13	3.00	4.02	1.64	2.93	2.22	3.34	4.10	1.90	2.25	2.92	2.86	3.22
2-8-13	2.61	2.30	1.55	2.90	2.79	4.02	2.79	2.48	1.45	3.24	4.10	4.10	2.42	2.10	1.91	2.15	3.20	2.24	3.81	2.14

METALLOGRAPHIC EXAMINATION

Metallographic sections of almost every combination of base and coating were prepared in the course of this investigation. Photomicrographs of a few of these sections and some general observations on the structures of the coatings are included. The photomicrographs illustrate the extreme irregularity of some of the coatings and indicate the magnitude of the problems involved in measuring these coatings with a magnetic gage. They may also help to explain the behavior of the coatings under exposure. For example, due to thick alloy layers some coatings may have rusty colored corrosion products long before there is any corrosion of the base metal.

TABLE X.—SUMMARY OF THICKNESS OF TWO ALUMINUM COATINGS ON ROUND RODS.

Basis Metal	Aluminum Coating	Magnetic Gage Reading, mils				Stripping Values	
		Average	Maximum	Minimum	Number of Readings	Mils	g per sq in.
1............	6	2.7	8+	1.7	20	5.0	0.224
	7	5.1	7.0	3.3	12	7.1	0.314
2............	6	3.4	8+	2.8	18	4.2	0.187
	7	3.9	4.6	3.2	18	5.2	0.230
4............	6	2.8	4.8	2.0	17	5.0	0.220
	7	2.6	3.3	1.9	19	5.4	0.240
0............	6	3.4	5.2	2.0	18	3.8	0.167
	7	4.5	6.7	3.3	18	4.5	0.273

Hot-Dip Aluminum Flat Panels:

The hot-dip aluminized coatings consisted of a layer of iron-aluminum alloy next to the steel surface covered by a layer of aluminum which contained a dispersion of iron-aluminum alloy crystals. Coating thicknesses tended to be somewhat nonuniform due to irregularities in the steel surface and, in some cases, a nonuniform attack of the steel by the aluminum. A complete study of the structures of the coatings was not carried out; however, it was noted that coating structures varied somewhat for the different steels compositions. In general, the thickness of the alloy layers in coating Code 7 was greater than in coating Code 6, suggesting that different processing techniques had been employed. Microstructures of some of the coatings are shown in Figs. 4 to 6.

Electroplated Zinc Flat Panels:

Two of the electroplated panels (basis metals 0 and 2) had fairly uniform coating thicknesses although the surfaces of the coatings were slightly irregular. The coating on the other panels (basis metal 1) was quite irregular due to the pronounced roughness of the steel surface and the low throwing power of the electroplating solution. This gave the surface of the panel a pitted appearance. Structures at typical areas are shown in Figs. 7 and 8.

Hot-Dip Zinc Flat Panels:

The structures of the galvanized coatings consisted of iron-zinc alloy layers on the steel surface covered by a zinc layer containing some iron-zinc alloy crystals. In general, the galvanized coatings are quite uniform.

The coatings on all of the steel panels (basis metals 0, 1, 2, 3, and 4) were quite similar in structure. All contained a fairly high proportion of alloy layers of

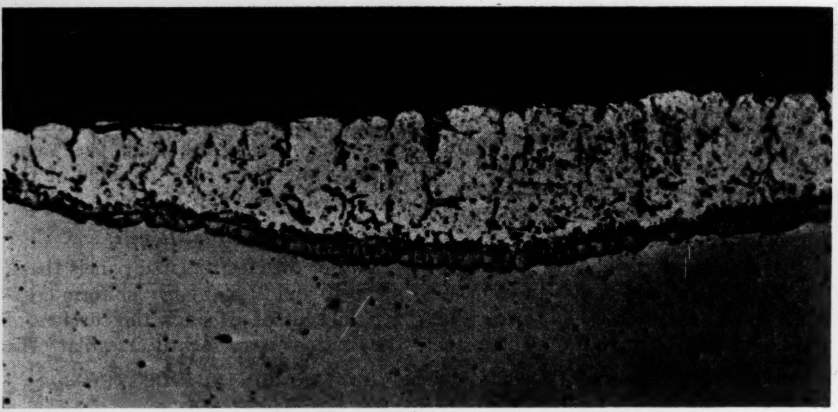


FIG. 4.—Structure of Hot-Dip Aluminum Coating No. 6 on Steel No. 2 (Code 2-6) (× 250).

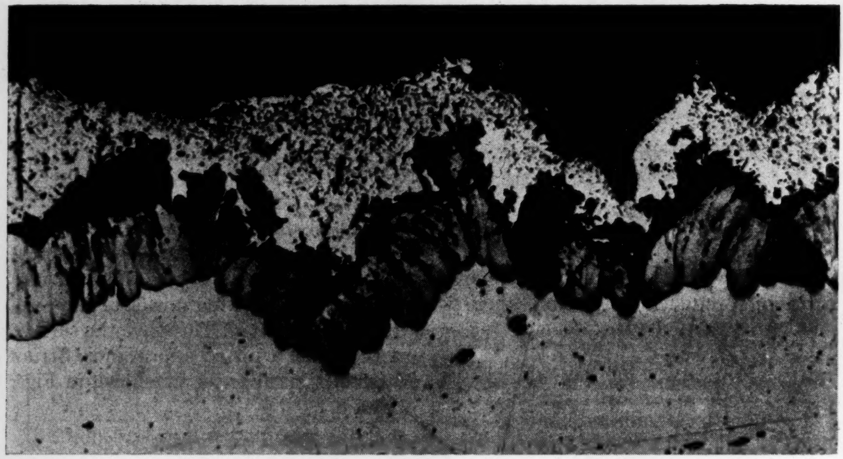


FIG. 5.—Structure of Hot-Dip Aluminum Coating No. 7 on Steel No. 2 (Code 2-7) (× 250).

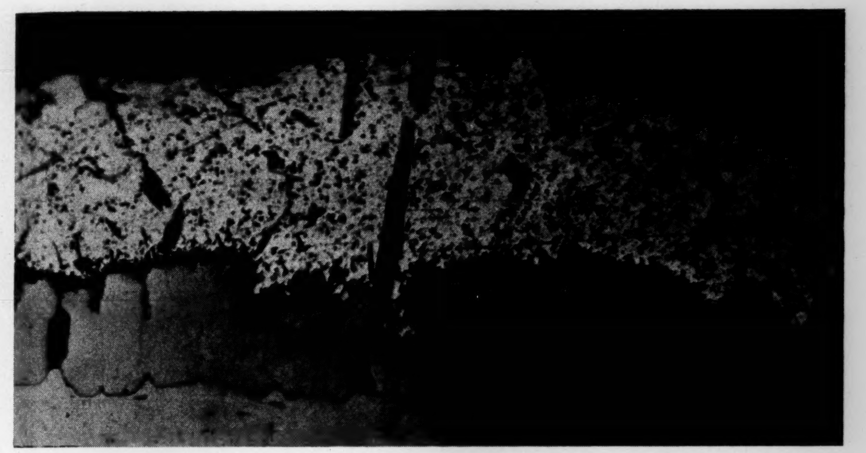


FIG. 6.—Structure of Hot-Dip Aluminum Coating No. 7 on Steel No. 0 (Code 0-7) (× 500).

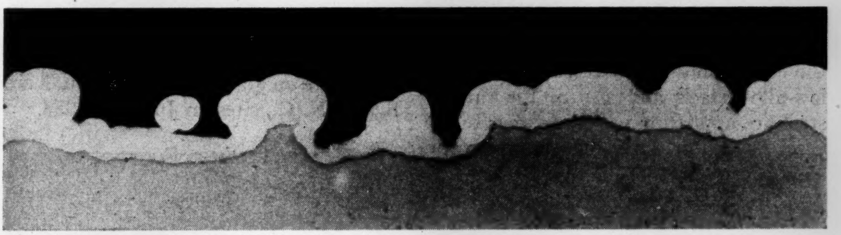


FIG. 7.—Electroplated Zinc Coating on Steel No. 1 Showing Nonuniform Thickness (Code 1-8) (× 100).

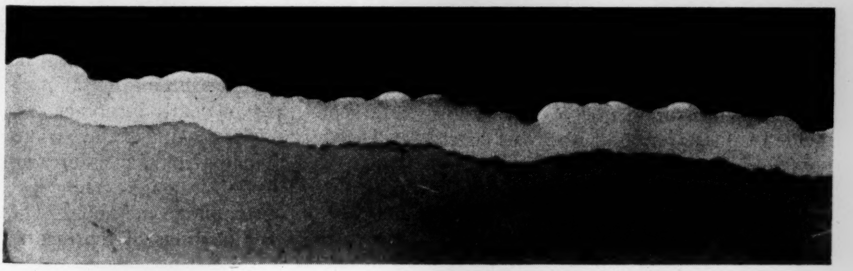


FIG. 8.— Electroplated Zinc Coating on Steel No. 2 (Code 2-8) (× 100).

normal structure. A typical section is shown in Fig. 9.

The coating on the malleable iron (basis metal 21) consisted almost entirely of iron-zinc alloy layers having a distinctive type of structure (see Fig. 10).

The nodular cast iron panels (basis metals 19 and 20) exhibited thick alloy layers with relatively thin zinc layers. On the machined panel (basis metal 20), graphite particles on the surface layers of the iron interfered with the alloying action resulting in pores in the coating (see Fig. 11). On the as-cast panel (basis metal 19), surface layers were free from graphite particles, and the coating was continuous.

Hot-Dip Aluminum Round Rods:

Figure 12 shows several cross-sections of a round rod of one low-alloy steel (III). The structure of the coating and the condition of the surface of the other low-alloy steels and also of the carbon steel are approximately the same. In all cases an outer layer of aluminum or aluminum-rich constituent was present. The thickness of this layer is not in any case uniform, varying between 0 and 4 mils. Minor gas pockets, large elongated voids, and separation of the duplex layer coating from the basis steel are noticeable. Fine radial and peripheral cracks can also be seen, some leading to elongated voids. In all cases, the cracks appear only in the iron-aluminum alloy. Hardness measurements of this alloy yielded values of around Rockwell hardness C 50 to C 60.

APPEARANCE OF FINISHED PANELS AND RODS

As has been mentioned before, the unevenness of many coatings is in part due to the heavily scaled and oxidized conditions of the basis metal surfaces. This is particularly noticeable in the round rods. The hot-dipped zinc panels and rods had some degree of surface roughness and localized buildup of coating metal. Many had streaks resulting from contacting one another during the galvanizing treatment. Some had black spots, probably due to incomplete scale removal during pickling. Those with the hot-dipped aluminum coating Code 6 had rough, uneven surfaces whereas those of coating Code 7 had, in general, a much smoother, even appearance. The electroplated zinc panels and rods had a smooth uniform coating, excepting the bent panels where the finish in most instances was shiny in the bent portion of the panel (low current density areas). The sprayed metal coatings, both zinc and aluminum, had a smooth appearance in both flat and bend panels.

EXPOSURE TIME

The exposure tests were begun as follows: At the New York Area (Newark, N. J.) site on June 13, 1958; at the Kure Beach, N. C., sites on June 23, 1958.

DISCUSSION AND CONCLUSIONS

Six types of coatings have been variously applied to carbon steel, several low-alloy steels, nodular and malleable iron. The panels and rods have been exposed to the industrial atmosphere at New York Area (Newark, N. J.) and to two marine atmospheres at Kure Beach, N. C. The progress of corrosion will be observed by visual examination at periodic semi-yearly or yearly intervals.

The thickness of coating of the various panels was evaluated by means of nondestructive magnetic gages and spot checked by stripping methods. The structure of the coatings of representative panels was examined by microsectioning. From an analysis of the collected data it is quite evident that the desired uniformity of coating thickness has not been obtained in all cases. The thicknesses of the majority of the hot-dipped zinc and hot-dipped aluminum coatings

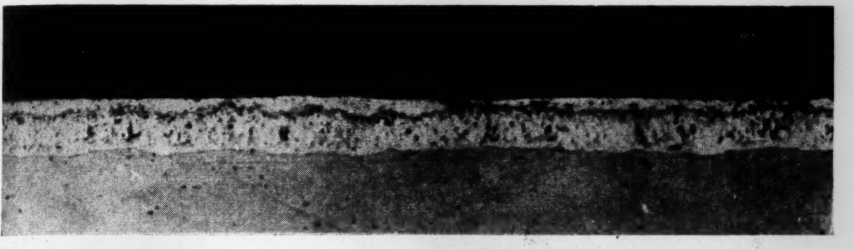


FIG. 9.—Structure of Hot-Dip Zinc Coating on Steel No. 3 (Code 3-5) (× 100).

FIG. 10.—Hot-Dip Zinc Coating on Malleable Iron (Code 21-5) (× 100).

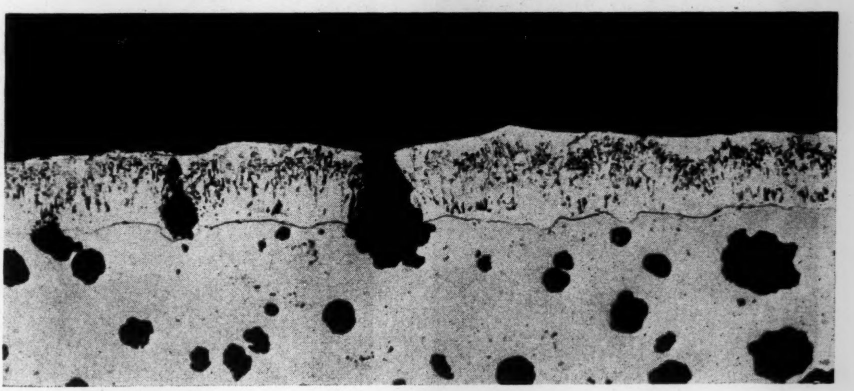


FIG. 11.—Coating on Machined Nodular Cast Iron Showing Pores in Zinc Coating Caused by Surface Graphite (Code 20-5) (× 100).

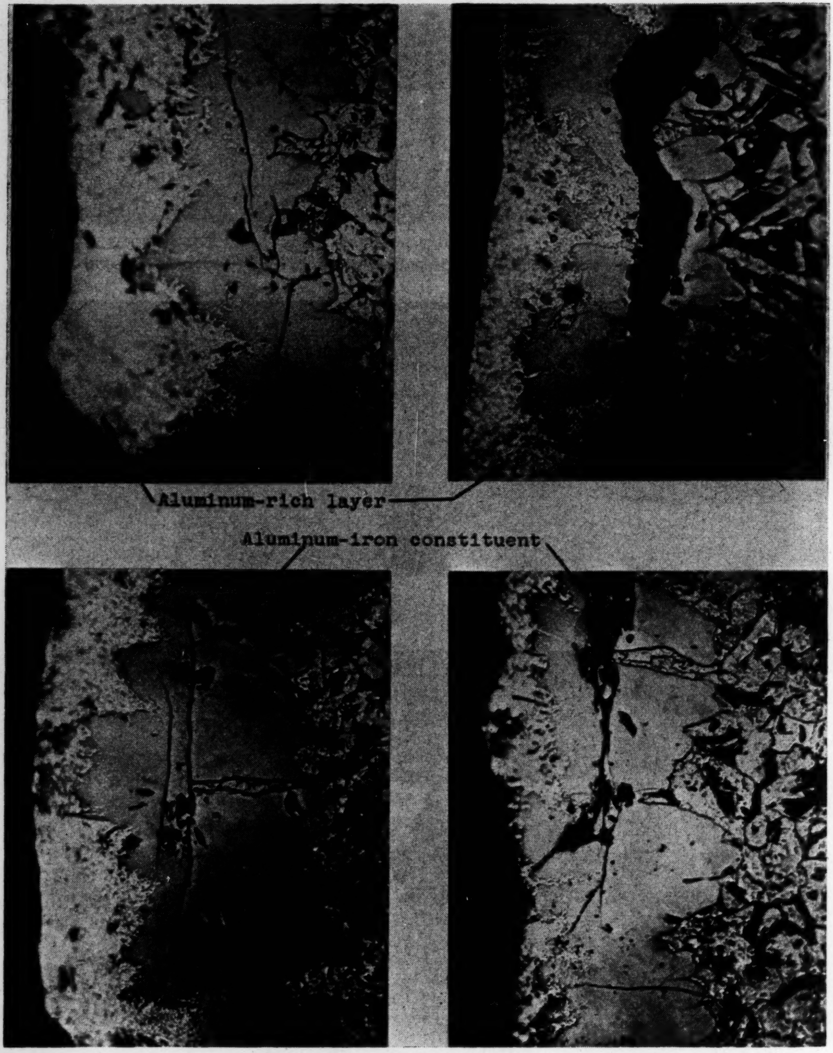


FIG. 12.—Photomicrographs Illustrating Various Portions of Outer Surface of Low Alloy Steel No. III (× 500).

on the flat and bent panels range from 1.5 to 4.0 mils. Individual measurements are as low as 1.4 and as high as 7.4 mils. Variations within a given group of five replicate samples are also significant. As for the coatings on the round rods, the thickness values given are open to question in view of the extremely uneven and heterogeneous coating. The voids and cracks in the alloy layer shown on the photomicrographs of the round rods suggest that they might have occurred during the cooling cycle. It is felt that these factors will have to be considered when evaluating the weathering capabilities of these coatings. In contrast to hot-dipped zinc coatings, the weight of coating determination of hot-dipped aluminum coatings by stripping is complicated by the fact that the aluminum-iron alloy has a density sufficiently different from that of the aluminum. Straight conversion of weight loss values to linear thicknesses is subject to error unless the exact ratio of aluminum to alloy is known. Judging from the accompanying photomicrographs (Figs. 4, 5, 6, 12), this may be a difficult task; it appears to be an impossible one for coatings on the round rods. However, regardless of whether these latter measurements are directly relatable to actual thickness values, they do indicate large differences in "apparent" coating thickness on individual rods as well as among the various lots of five panel and rod sets.

Respectfully submitted on behalf of the subcommittee,

A. MENDIZZA,
Chairman.

REPORT OF COMMITTEE A-6
ON
MAGNETIC PROPERTIES*

Committee A-6 on Magnetic Properties held two general meetings during the year: on June 24, 1958, at the Annual Meeting of the Society in Boston, Mass., and on Feb. 5, 1959, in Pittsburgh during ASTM Committee Week.

Two very faithful workers retired during the year: G. H. Cole, who represented the Armco Steel Corp. for many years, and A. A. Frey, who similarly represented the Wheeling Steel Corp. Another member who contributed much to the work of this committee, K. V. Merrill of the Niles Rolling Mill Division of the Sharon Steel Corp., died suddenly on December 9, 1958.

The basic organization of the committee remains the same as it was at the time of the last published report, June 1958. The representation of Committee A-6 on other ASTM committees and on committees of other standardizing organizations is as follows:

ASTM Committee E-8 on Nomenclature and Definitions, I. L. Cooter.
ASTM Committee E-11 on Quality Control of Materials, D. J. Knight.
ASTM Committee C-21 on Ceramic Whitewares and Related Products, Subcommittee II on Tests and Specifications, Section A-5 on Ferrites, A. C. Beiler.
American Standards Association, Sectional Committee C42 on Definitions of Electrical Terms, I. L. Cooter.
American Standards Association, Sectional Committee C61 on Electric and Magnetic Magnitudes and Units, I. L. Cooter.
International Electrotechnical Commission, Subcommittee 14-A on Magnetic Steel, Technical Advisor to U. S. National Committee, A. C. Beiler.
American Society of Tool Engineers, Project on Metal Stamping, G. H. Cole, until Dec. 1, 1958. No successor named to date.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (I. L. Cooter, chairman) is continuing revision of the magnetic definitions in an effort to maintain uniformity between ASTM and the American Standard proposed by ASA Sectional Committee C42. At the present time, the major portion of this revision has been completed and it is anticipated that a complete revision of Definitions of Terms, with Symbols, Relating to Magnetic Testing (A 340 – 49) will be available before the end of 1959.

Subcommittee II on Methods of Sampling and Test (J. W. Hale, chairman) has prepared a preliminary revision of the Standard Methods of Test for Alternating Current Core Loss and Permeability of Magnetic Materials (A 343 – 54). At the present time, this proposal has no official status as a tentative specification, but it has been printed and copies are available on request from Society Headquarters in order to provide a basis for discussion and further revision.

Under the leadership of Mr. A. E. Kettner, additional work by one of the task groups has resulted in a tentative revision of the Standard Methods of Test for Permeability of Feebly Magnetic Materials (A 342 – 53) which includes a new test method developed by the Naval Ordnance Laboratory. After editorial corrections and submission to the main committee, this revision will probably be available later in 1959.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

Another task group, also under the leadership of Mr. A. E. Kettner, is making extensive revisions of the Standard Methods of Test for Electrical and Mechanical Properties of Magnetic Materials (A 344 – 52). While the revisions are general, the chief revisions have to do with test methods for measurements of laminations stack resistance and of surface insulation resistance.

Subcommittee III, Editorial (C. C. Horstman, chairman) has followed up the work of Subcommittee II and is making a revision of Methods A 343 mentioned above.

In view of the revision of Methods A 342 and A 344, it is presumed that the task groups working on interlaminar resistance tests and on low permeability tests, both of which were under Mr. A. E. Kettner's leadership, can now be dissolved. Task groups, largely under Subcommittee II, which are still active are listed below with their leaders:

1. Manual of Testing (C. A. Maynard),
2. Effect of Aluminum Additions on Density of Silicon-Iron Alloys (D. H. Jones),
3. Aluminum-Iron Density Tables (D. I. Gordon),
4. Magnetic Core Materials (W. T. Mitchell),
5. Method of Test for Cut Tape-Wound Cores (E. W. Hill),
6. D-C Testing of High Coercive Magnets (C. A. Maynard), and
7. Methods of Testing Magnetic Shielding (W. I. Lewis).

This report has been submitted to letter ballot of the committee, which consists of 28 voting members; 20 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. Beiler,
Chairman.

W. S. Eberly,
Secretary.

REPORT OF COMMITTEE A-10
ON
IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS*

Committee A-10 held two meetings during the year: at Boston, Mass., in June 1958, during the Annual Meeting of the Society, and at Pittsburgh, Pa., in February 1958, during ASTM Committee Week.

The committee consists of 115 members, of whom 52 are classified as producers, 42 as consumers, and 21 as general interest members.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentative specifications and their continuation as tentative:

Tentative Specifications for Corrosion-Resisting Chromium Steel Clad Plate, Sheet, and Strip (A 263 - 44 T):[1]

Section 3.—Replace with the following:

3. Unless otherwise specified or agreed upon between the purchaser and the manufacturer, all plates shall be furnished in the normalized and tempered condition.

Tentative Specifications for Corrosion-Resisting Chromium - Nickel Steel Clad Plate, Sheet, and Strip (A 264 - 44 T):[1]

Section 3.—Replace with the following:

3. (*a*) Unless otherwise specified or agreed upon between the purchaser and the manufacturer, all austenitic stainless steel clad plates shall be given a heat treatment consisting of heating to the proper temperature for the solution of the chromium carbides in the cladding followed by individual air cooling. For base metals of air hardening low alloy steels the above heat treatment shall be followed by a tempering treatment.

(*b*) When plates over 1 in. in thickness are to be cold formed the purchaser may specify that such plates be heat treated for grain refinement of the base metal.

Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 58 T):[1]

Revise as indicated in the 1959 Report of Committee A-1 on Steel.[2]

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specifications for Hot-Rolled and Cold-Finished Corrosion-Resisting Steel Bars (**A 276 - 57 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] 1958 Book of ASTM Standards, Part 1.

[2] This revision has been recommended by a Joint A-1 and A-10 Special Subcommittee on Stainless Steel Tubing. It appears in the 1959 Supplement to Book of ASTM Standards, Part 1.

Standard Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 58):[1]

Revise as indicated in the 1959 Report of Committee A-1 on Steel.[2]

Standard Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 58):[1]

Revise as indicated in the 1959 Report of Committee A-1 on Steel.[2]

Standard Specifications for Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 - 55):[1]

Table II.—Add grade CA-40, with the following chemical requirements:

Carbon, per cent	0.20 to 0.40
Manganese, max, per cent	1.00
Silicon, max, per cent	1.50
Phosphorus, max, per cent	0.05
Sulfur, max, per cent	0.05
Chromium, per cent	11.5 to 14.0
Nickel, max, per cent	1.0
Molybdenum, max, per cent	0.5

Change the silicon content of grade CB-30 from "1.00" to "1.5 max."

Change the silicon content of grade CC-50 from "1.00" to "1.5 max."

Table III.—Add grade CA-40, with the following tensile requirements:

Tensile strength, min, psi	90,000
Yield strength, min, psi	65,000
Elongation in 2 in., min, per cent	18
Reduction of area, min, per cent	30

Standard Specifications for Heat-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 297 - 55):[1]

Table II.—Change the minimum elongation of grade HH from "15" to "10" per cent. Change the minimum tensile strength and the minimum elongation of grade HI from 75,000 psi and 15 per cent to 70,000 psi and 10 per cent respectively. Change the minimum tensile strength and the minimum elongation of grade HK from 75,000 psi and 15 per cent to 65,000 psi and 10 per cent respectively.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tenative Recommended Practice for:

Boiling Nitric Acid Test for Corrosion-Resisting Steels (**A 262 – 55 T**), and
Conducting Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steel (**A 393 – 55 T**).

Tentative Method of:

Total Immersion Corrosion Test for Stainless Steels (**A 279 – 44 T**).

Tentative Specifications for:

Nickel and Nickel-Base Alloy Clad Steel Plate (**A 265 – 43 T**),
Iron-Chromium and Iron-Chromium-Nickel Alloy Tubular Centrifugal Castings for General Applications (**A 362 – 52 T**), and
Corrosion-Resisting Chromium-Nickel-Manganese Steel Plate, Sheet, and Strip (**A 412 – 57 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee IV on Methods of Corrosion Testing (O. B. Ellis, chairman).—The task force under the chairmanship of F. K. Bloom which is preparing the comprehensive report on stress corrosion cracking expects to have its work completed by the 1959 Annual Meeting. This report has been called to the attention of the Administrative Committee on Paper and Publications for consideration of its publication by the Society as a Special Technical Publication.[4]

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The program for Atmospheric Corrosion Tests, under the task group headed by C. R. Mayne, has completed the placing of its test panels; the details of the placement and materials are shown in Table I.

The Task Force on Cleaning of Stainless Steel (E. G. Holmberg, chairman) has reported that in Recommended Practice A 380 the use of aceto-acetanilide solution to detect iron contaminants is unsatisfactory; steps are being taken to delete this procedure from the recommended practice.

The Task Force on Intergranular Corrosion Tests (W. G. Renshaw, chairman) has reported the continuation of round-robin tests; a progress report may be expected by the 1959 Annual Meeting.

Subcommittee V on Mechanical Tests (R. H. Heyer, chairman).—The Task Group on Hardness Testing (A. M. Hall, chairman) is continuing its work on the Brinell and Rockwell B hardness tests on 4 by 8½ in. test blocks of eight plate materials from ⅜ to 1 in. thick on eight grades of steel of Brinell hardness of 120 to 220 in order to obtain conversion test data for these materials.

The new Subcommittee V–IX Task Group on Tension Testing of Thin Sheets (A. G. Cook, chairman) has already distributed test methods in use by several laboratories and is actively engaged in studying these methods and their significance.

In addition, the subcommittee has discussed the subject of standard specimens for stress-rupture and creep tests, and arrangements are being made for adequate liaison representation with the Testing Panel of the Joint Committee on Effect of Temperature on Properties of Metals.

Subcommittee VI on Metallography (Russell Franks, chairman).—Two task groups are currently working on the metallographic aspects of stainless steels: the first, with J. J. Heger as chairman, is concerned with the identification of finely dispersed microconstituents following the sensitization of type 316 steel; the second, with F. L. Ver Snyder as chairman, is concerned with the causes of cracking in welded type 347, and is also compiling a case history of

TABLE I.—SPECIMENS FOR ATMOSPHERIC CORROSION TEST.

Place	Date	Specimens
Kure Beach, N. C. (800-ft lot)	May 27, 28 and June 2, 1958	732
Newark, N. J.	June 17, 1958	731
State College, Pa.	July 8, 1958	732
Brazos River, Tex.	July 21, 1958	732
Kure Beach, N. C. (80-ft lot)	October 1, 1958	635
Battelle Memorial Inst., Columbus, Ohio	November 14, 1958	622

NOTE.—Types of material included in this program: 410, 416, 430, 301 (annealed, quarter hard, half hard, three quarter hard), 302 (annealed, quarter hard), 303, 303Se, 304, 304L, 316, 316L, 321, 347, 201 (annealed, quarter hard, half hard, three quarter hard), 202, 16-16-1 (annealed, half hard), and 16-9-5 (0.03 max carbon). Specimens exposed included: spot welded panels, arc welded panels, tension panels, Erikson cup panels, 0.505-in. tension test specimens, appearance panels, and springs for making fatigue tests. The first removal will be in 1961 and subsequent removals in 7 and 15 years.

---

[4] To be issued as separate publication *ASTM STP No. 264*.

samples which have reacted both favorably and unfavorably when welded.

Subcommittee VIII on Specifications for Wrought Products (R. B. Gunia, chairman).—This subcommittee has completed its review of the specifications under its jurisdiction and is currently directing its attention toward the necessity for standards concerning forged or extruded products, as well as toward the various age-hardening types of stainless steels.

Subcommittee IX on Specifications for Flat Products (G. W. Hinkle, chairman). —The subcommittee has been giving consideration to Specifications A 167, A 176, and A 240; it appears that the latter will need revision, particularly with respect to the addition of H grades. A task force (A. G. Cook, chairman) has been appointed to consider and make recommendations on the 0.04 to 0.10 per cent carbon modification of the H grades.

Following general agreement on the desirability of having a general purpose specification for flat-rolled products, a task group (R. Sergeson, chairman) was appointed to develop the specification.

In connection with the recommendations of the joint A-1 and A-10 group headed by C. L. Clark, Subcommittee IX is recommending the adoption of new heat-treatment clauses in Specifications A 263 and A 264.

Subcommittee X on Specifications for Castings (H. C. Templeton, chairman). —Due to the press of other duties, J. J. Kanter considered it necessary to relinquish his long chairmanship of the subcommittee; his resignation was accepted with reluctance by the Advisory Committee, which expressed the sincere appreciation of Committee A-10 for his services. Mr. H. C. Templeton was appointed to fill the vacancy.

The subcommittee has reaffirmed the several changes in Specifications A 296 which inadvertently escaped publication; full committee action will be taken and reported in the ASTM BULLETIN. By several actions of the subcommittee, new grades CF-8, CF-3, CF-3M, CG-8M, HD, HL, and HN are to be recommended for inclusion in Specifications A 296; the letter ballots are currently being circulated.

The subcommittee also approved additional study by the joint task group concerned with types CF- and CF-M.

Subcommittee XI on Specifications for Tubular Products (J. J. B. Rutherford, chairman).—This subcommittee, which acts in conjunction with Subcommittee IX of Committee A-1, held a joint meeting with Committee A-1; the actions taken and recommendations are noted in the report of Committee A-1. The several items requiring concurrent action in Committee A-10 (Specifications A 269, A 271, and A 312) are subject to current letter ballot. The revisions proposed for Specifications A 213 and A 249 in the report of Committee A-1 were also considered by this subcommittee.

Subcommittee XII on Specifications for High-Temperature, Super-Strength Alloys (W. F. Simmons, chairman).—Both Subgroup A on Hot-Cold Worked Alloys (C. L. Clark, chairman) and Subgroup B on Precipitation Hardened Alloys (M. C. Metzger, chairman) have submitted drafts of proposed tentative specifications for alloys within the respective groups. These are now being reviewed preparatory to discussion and final drafting.

The subcommittee has also authorized the revision of STP 170A, "A Compilation of Chemical Compositions and Rupture Strengths of Super-Strength Alloys." This revision should be completed in 1959.

Subcommittee XIII on Specifications for Nuclear Reactor Structural Materials (M. A. Cordovi, chairman).—The subcommittee has six highly active task groups engaged in surveying areas of desired activity. In general, it is considered the more advisable policy to amplify existing specifications rather than to write specialty items.

Of the items of current interest, much consideration is being given to the tolerable cobalt limits (0.05 to 0.20 per cent), and to the need for spectrographic standards and wet analytical procedures for cobalt, boron, and rare earths.

The task groups will, where considered necessary, prepare "Special (Optional) Requirements for Nuclear Service" where applicable for addenda to existing Committee A-10 specifications.

This report has been submitted to letter ballot of the committee, which consists of 115 members; 84 members returned their ballots, of whom 80 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

L. B. FONDA,
Secretary.

REPORT OF COMMITTEE B-1
ON
WIRES FOR ELECTRICAL CONDUCTORS*

Committee B-1 on Wires for Electrical Conductors held three meetings during the year: in Washington, D. C., on October 24, 1958; in New York, N. Y., on January 20, 1959; and in Philadelphia, Pa., on April 10, 1959. Meetings of Subcommittees II, IV, V, and VII also were held during the year.

During the year B. J. Sirois resigned the chairmanship of Subcommittee IV, due to a change in work assignment, and M. V. Yokelson was appointed to this office. The committee took action to record its appreciation of Mr. Sirois' past services.

The committee consists of 86 members, of whom 44 are classified as producers, 27 as consumers, and 15 as general interest members.

NEW TENTATIVES

The committee recommends for publication as tentative the following method and specifications as appended hereto:[1]

Tentative Method of Test for Electrical Conductivity by Use of Eddy Currents:

About two years ago a request was received from Committee B-7 for the inclusion of the eddy current method of measuring resistivity in Method B 193. Although this method has a lower order of reliability than the bridge method now included in B 193 and is not applicable to round wires (hence not usable in conjunction with most of the Committee B-1 product specifications), it was felt that since this method is being used commercially on certain conductor materials such as bus bars, a formal procedure should be prepared under the jurisdiction of Committee B-1. Accordingly, a separate method of test is recommended, including notations on differences in applicability between it and B 193.

Tentative Specifications for Aluminum-Coated (Aluminized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR):

During the past few years there has been growing commercial interest in aluminized-steel core wire for steel-reinforced aluminum conductors. About three years ago the committee took note of this trend and started developing suitable specification requirements, as part of its continuing program of developing proposed specifications for several particular types of aluminum conductors. Accordingly, the new proposed specification is recommended to supplement the present specifications for galvanized-steel core wires.

REVISIONS OF TENTATIVES

The committee recommends revisions

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 2.

TABLE I.—ADDITIONS TO TABLE I, SPECIFICATIONS B 232.

Conductor Size (Aluminum)		Stranding			
		Aluminum		Steel	
cir mils	Awg	Number of Wires	Diameter, in.	Number of Wires	Diameter, in.
1 590 000....	...	45	0.1880	7	0.1253
1 431 000....	...	45	0.1783	7	0.1189
1 272 000....	...	45	0.1681	7	0.1121
1 192 500....	...	45	0.1628	7	0.1085
1 113 000....	...	45	0.1573	7	0.1048
954 000....	...	45	0.1456	7	0.0971
795 000....	...	45	0.1329	7	0.0886

TABLE II.—ADDITIONS TO TABLE V, SPECIFICATIONS B 232.

Conductor Size, cir mils or Awg	Stranding		Weight per 1000 ft, lb	Nominal Length of Each Piece, ft	Nominal Weight of Each Length, lb
	Aluminum	Steel			
1 590 000............	45 × 0.1880	7 × 0.1253	1792	2390	4283
1 431 000............	45 × 0.1783	7 × 0.1189	1613	2660	4291
1 272 000............	45 × 0.1681	7 × 0.1121	1434	2990	4288
1 192 500............	45 × 0.1628	7 × 0.1085	1344	3190	4287
1 113 000............	45 × 0.1573	7 × 0.1048	1255	3420	4292
954 000............	45 × 0.1456	7 × 0.0971	1075	3990	4289
795 000............	45 × 0.1329	7 × 0.0886	896	4790	4292

as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (ACSR) (B 232 – 58 T):[2]

Section 1(a).—Revise to read "1.(*a*) These specifications cover concentric-lay-stranded conductors made from round aluminum wires and round zinc-coated or aluminum-coated steel core wires for use as . . . ."

Section 3(b).—Change to read as follows:

(*b*) Before stranding, the steel core wires used shall meet all of the requirements of the following specifications that are applicable to its type: Specifications for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (ASTM Designation: B 245);[3] Specifications for Zinc-Coated (Galvanized) Steel Core Wire (With Coatings Heavier than Standard Weight) for Aluminum Conductors, Steel Reinforced (ACSR) (ASTM Designation: B 261);[3] and Specification for Aluminum-Coated (Aluminized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (ASTM Designation: B 341).[3]

Table I.—Revise by the addition of the conductors shown in the accompanying Table I. Delete the conductors of 1,510,500; 1,351,500; 1,033,500; and 874,500 cir mils.

Section 7.—Add a new Paragraph (*c*) to read as follows:

(*c*) When requested by the purchaser at the time of placing the order, tension tests on wires before stranding or on the completed conductor as a unit may be waived and tests made on aluminum wires removed from the completed ACSR conductor. When so tested, hard-drawn wires shall have minimum tensile strengths not less than 95 per cent of the tensile strengths prescribed for individual tests in Table I of ASTM Specifications B 230 (Note 7).

Reletter the present Paragraph (*c*) as (*d*) and change to read: "(*d*) Tests for all properties of zinc- or aluminum-coated steel wires shall be made before stranding (Note 7)."

Table IV.—Insert the following between the ninth and tenth lines of the table:

---

[2] 1958 Book of ASTM Standards, Part 2.

Stranding of ACSR, Number of Wires		Increment (Increase), per cent		
		Weight		Electrical Resistance
Aluminum	Steel	Aluminum	Steel	
45......	7......	2.5	0.4	2.5

Table V.—Revise by the addition of the conductors shown in the accompanying Table II. Delete the conductors of 1,510,500; 1,351,500; 1,033,500; and 874,500 cir mils.

Section 13.—In the first sentence, delete the parenthetical clause "(or lengths and number of pieces, if more than one length is included in a package)."

Explanatory Note 7.—Change the second sentence to read: "If tests on galvanized or aluminum-coated steel wires are to be made after stranding, the purchaser and the manufacturer at the time of placing the order should agree on the properties to be met."

Tentative Specifications for Soft or Annealed Coated Copper Conductors for Use in Hookup Wire for Electronic Equipment (B 286 – 57 T):[2]

Title.—Delete the words "Soft or Annealed Coated."

Section 1.—Delete the words "soft or annealed."

Section 2.—Renumber Item (*3*) as (*5*). Renumber Item (*4*) as (*3*) and change to read: "Whether tin, lead-alloy, or silver-coated (Section 3(*a*))." Add a new Item (*4*) to read: "(*4*) For silver-coated conductors, class of coating (Section 3(*a*)), and when required, unannealed (Section 3(*b*))." Renumber Items (*5*), (*6*), and (*7*) as (*6*), (*7*), and (*8*) respectively.

Section 3.—At the end of Paragraph (*a*) add: "Specifications for Silver-Coated Soft or Annealed Copper Wire (ASTM Designation: B 298)."

TABLE III.—REVISION OF TABLE II, SPECIFICATIONS B 286.

TYPE I (SOLID CONDUCTORS)

Size Designation, Awg	d-c Resistance at 20 C, ohms per 1000 ft, max (Note 1)	
	Annealed Tin or Lead-Alloy Coated	Annealed Silver-Coated
10..............	1.06	1.02
12..............	1.69	1.62
14..............	2.68	2.58
16..............	4.26	4.10
18..............	6.78	6.52
20..............	10.7	10.3
22..............	17.2	16.5
24..............	27.2	26.2
26..............	44.5	41.9

TYPE II (STRANDED CONDUCTORS)

Size Designation[d]	d-c Resistance at 20 C, ohms per 1000 ft, max (Note 1)	
	Annealed Tin or Lead-Alloy Coated	Annealed Silver-Coated
0000-2109........	0.0577	0.0537
000-1672........	0.0728	0.0678
00-1330........	0.0915	0.0852
0-1064........	0.114	0.107
1-836.........	0.146	0.136
2-665.........	0.181	0.169
4-420.........	0.287	0.267
6-266.........	0.453	0.422
8-168.........	0.717	0.668
10-104.........	1.14	1.06
12-65..........	1.82	1.69
12-19[c]..........	1.92	1.81
14-41..........	2.88	2.69
14-19[c]..........	3.05	2.87
16-26..........	4.55	4.23
16-19[c]..........	4.82	4.54
18-26[c]..........	7.10	6.62
18-19[c]..........	6.22	5.80
18-7[c]	6.61	6.22
20-19[c]..........	9.72	9.05
20-10..........	11.8	11.0
20-7..........	10.5	9.9
22-19[c]..........	15.7	14.6
22-7[c]..........	16.9	15.7
24-19[c]..........	24.9	23.2
24-7[c]	26.4	24.6
26-7[c]..........	42.5	39.6
28-7[c]..........	67.5	62.9
30-7[c]..........	106.0	98.0
32-7[c]..........	176.0	164.0

[d] These size designations are solely for purposes of identification. They should not be confused with Awg sizes.

TABLE IV.—REVISION OF TABLE III, SPECIFICATIONS B 286.

TYPE I (SOLID CONDUCTORS)

Size Designation, Awg	d-c Resistance at 20 C, ohms per 1000 ft, max		Size Designation, Awg	d-c Resistance at 20 C, ohms per 1000 ft, max	
	Annealed Tin or Lead-Alloy Coated	Annealed Silver-Coated		Annealed Tin or Lead-Alloy Coated	Annealed Silver-Coated
10	1.10	1.06	20	11.2	10.7
12	1.75	1.69	22	17.9	17.2
14	2.79	2.68	24	28.3	27.2
16	4.44	4.27	26	46.2	43.5
18	7.05	6.78			

TYPE II (STRANDED CONDUCTORS)

Size Designation[a]	d-c Resistance at 20 C, ohms per 1000 ft, max		Size Designation	d-c Resistance at 20 C, ohms per 1000 ft, max	
	Annealed Tin or Lead-Alloy Coated	Annealed Silver-Coated		Annealed Tin or Lead-Alloy Coated	Annealed Silver-Coated
0000-2109	0.060	0.0559	18-26	7.39	6.88
000-1672	0.0757	0.0705	18-19	6.47	6.03
00-1330	0.0951	0.0886	18-7	6.87	6.47
0-1064	0.119	0.111	20-19	10.1	9.42
1-836	0.151	0.141	20-10	12.3	11.5
2-665	0.188	0.176	20-7	10.9	10.3
4-420	0.298	0.278	22-19	16.3	15.2
6-266	0.471	0.439	22-7	17.6	16.4
8-168	0.746	0.695	24-19	25.9	24.1
10-104	1.18	1.10	24-7	27.4	25.6
12-65	1.89	1.76	26-7	44.2	41.2
12–19	2.00	1.88	28-7	70.2	65.4
14-41	3.00	2.79	30-7	110.0	102.0
14-19	3.17	2.99	32-7	183.0	170.0
16-26	4.73	4.40			
16-19	5.07	4.72			

[a] These size designations are solely for purposes of identification. They should not be confused with Awg sizes.

Add a new Paragraph (*b*) to read as follows, relettering subsequent paragraphs accordingly:

(*b*) *Temper.*—Unless otherwise specified all coated conductors shall be furnished in the annealed temper. When so specified, silver-coated conductors shall be furnished unannealed (Note 1). (NOTE.—The term unannealed as used in this specification means cold-worked conductor as produced on commercial wire-drawing machines.)

Reletter the present Paragraphs (*b*) and (*c*) as (*c*) and (*d*) respectively and revise to read as follows:

(*c*) *Tensile Strength and Elongation.*—The tensile strength and elongation of annealed solid and of individual wires removed from stranded conductors shall conform to the values prescribed in Table I.

(*d*) *D-C Resistance.*—The d-c resistance in ohms per 1000 ft of annealed solid and stranded conductor shall not exceed, before insulating, the appropriate values prescribed in Table II (Note 2).

Section 5(*c*).—Change "(Note 2)" to read "(Note 3)."

Section 6.—Change "(Note 3)" to read "(Note 4)."

Section 7.—In Paragraph (*a*) change

the words "either ASTM Specifications B 33 or B 189" to read "ASTM Specifications B 33, B 189, or B 298."

In Paragraph (*c*) change "(Note 1)" to read "(Note 2)."

Table II.—Delete the present right-hand column and replace with the double column, for both Type I and Type II conductors, as shown in the accompanying Table III.

Section 8.—Add the word "coated" before the word "copper." Change "(Note 4)" to read "(Note 5)."

Explanatory Notes.—Add a new Note 1 to read as follows, renumbering subsequent notes accordingly. "NOTE 1.—Unannealed silver-coated conductors should be used only when the insulating process will produce an annealed insulated conductor."

Renumber the present Note 1 as 2 and insert the following sentence between the third and fourth sentences: "The values appearing in Table III under the heading "Annealed Silver-Coated" are applicable to silver-coated conductors whether annealed or unannealed prior to insulating."

In the second paragraph of Note 1, delete the definition of ρ and insert the following table in its place:

Type of Wire	Range of Wire Sizes, in.	ρ, ohm-pound/mile^2
Annealed silver-coated	all	875.20
Annealed tin or lead-alloy coated	up to 0.0110, incl	939.51
	over 0.0110 to 0.0200, incl	929.52
	over 0.0200	910.15

Revise the definition of stranding lay actor "f = 1.02" to read "f = 1.02 for bunch- and concentric-stranded conductors." Revise the second sentence in the definition of stretch allowance factor m to read "For solid conductors, the value of m therefore is 1.00, but for bunch-, concentric-, and rope-lay-stranded conductors m is taken as 1.02," and at the end of the last paragraph delete "B 33 and B 189" and replace with "B 33, B 189, and B 298."

Table III.—Delete the present resistance-value columns and replace with the double columns shown in the accompanying Table IV, for both type I and type II conductors.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

TABLE V.—MAXIMUM LENGTH OF LAY FOR BUNCH-STRANDED CONDUCTORS.
(Revision of Table II, Specifications B 174)

Size, Awg	Nominal Diameter, in.	Nominal Area, cir mils	Maximum Length of Lay, in.	
			Column A	Column B[a]
7......	0.167	20820	3.00	3.00
8......	0.149	16510	2.75	2.75
9......	0.133	13090	2.50	2.50
10......	0.118	10380	2.50	2.50
12......	0.093	6530	2.00	2.00
14......	0.074	4110	2.00	1.62
16......	0.059	2580	2.00	1.25
18......	0.047	1620	2.00	1.00
20......	0.037	1020	2.00	1.00

[a] The lengths of lay of column B meet the requirements of the following types of flexible cords and fixture wires as defined by Underwriters' Laboratories, Inc.: types S, SJ, SO, SJO, SJT, ST, SV, SVT, SF, SFF, SP, SPT, and HPN for sizes 10 Awg through 18 Awg and for types FX, FXT, X, and XT for size 20 only.

Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 - 56):[2]

Section 11(a).—Revise the first sentence to read: "The area of cross-section of the completed conductor shall be not less than 98 per cent of the area of cross-section listed in column 1 of Table II."

Standard Specifications for Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 - 58):[2]

Table I.—Delete the fourth column, Typical Uses. Insert (in 7 places) references to footnote *b* after "20" in column two, Conductor Sizes, Awg.

Delete the present footnote *b* and replace with the following:

[b] Size 20 is restricted in the schedule of requirements by Underwriters' Laboratories, Inc., to usages such as types FX, FXT, X, and XT and does not meet Underwriters' Laboratories, Inc., requirements for fixture wires unless such usage has been approved for specific applications.

Table II.—Change Table II to read as shown in the accompanying Table V.

Section 6.—Change Paragraph (*c*) to read: "(*c*) Unless otherwise specified by the purchaser, the length of lay of bare or coated bunch-stranded conductors shall conform to the requirements of column B of Table II."

Change Paragraph (*d*) to read: "(*d*) When specified, for constructions other than those listed in footnote *a* of Table II, the length of lay shall conform to the requirements of column A of Table II."

Change Paragraph (*e*) to read: "(*e*) Conductors of an intermediate nominal area in circular mils shall conform to the requirements for length of lay of the next smaller conductor."

Add a new Paragraph (*f*) to read as follows:

(*f*) Underwriters' Laboratories, Inc. requirements state that uncoated conductors which are to be rubber or neoprene insulated, and all conductors having lengths of lay longer than those specified by column B of Table II shall be provided with a separator between the conductor and the insulation.

Section 8.—In Paragraph (*d*) change "(Note 4)" to read "(Note 3)." In Paragraph (*f*) change "(Note 5)" to read "(Note 4)."

Section 9.—Change "(Note 6)" to read "(Note 5)."

Explanatory Notes.—Delete Note 3 and renumber subsequent notes accordingly.

Standard Method of Test for Resistivity of Electrical Conductor Materials (B 193 - 58):[3]

Table II.—In the listing under Aluminum Alloy GS10B, per cent IACS, revise as follows:

Present	Revise to
57	57.0
56	56.0
55	55.0

(This editorial change is made to agree with similar data in Specification B 317 - 58 T).

Standard Specifications for Concentric-Lay-Stranded Aluminum Conductors, Hard, Three-Quarter Hard and Half-Hard (B 231 - 58):[2]

Section 7(c).—Change to read as follows:

(*c*) When requested by the purchaser at the time of placing the order, tension tests on wires before stranding or on the completed conductor as a unit may be waived and tests made on wires removed from the completed conductor. When so tested, hard-drawn wires shall have minimum tensile strengths no less than 95 per cent of the tensile strengths prescribed for individual tests in Table I of the Specifications for Hard-Drawn Aluminum Wire for Electrical Purposes (ASTM Designation: B 230), and three-quarter hard or half-hard wires shall have tensile strengths not less than 95 per cent of the minimum tensile strength nor more than 105 per cent of the maximum tensile strength prescribed in Section 5(*a*) of the Specifications for Three-Quarter Hard Aluminum Wire for Electrical Purposes (ASTM Designation: B 262) or Section 5(*a*) of the Specification for Half-Hard Aluminum Wire for Electrical Purposes (ASTM Designation: B 323) (Note 5).

---

[3] 1958 Book of ASTM Standards, Part 3.

Section 11.—Change the first sentence to read: "The area of cross-section of the completed conductor shall be not less than 98 per cent of the area of cross-section or conductor size listed in column 1 of Table II."

Standard Specifications for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 245 – 58):[2]

Section 5(c).—In both Item (*1*), *Nonautographic Extensometer*, and Item (*2*), *Autographic Extensometer*, change the first word "If" to read "When."

Section 7(a).—In the second sentence, change ". . . galvanized ACSR wire . . ." to read ". . . galvanized steel core wire . . ."

Table VI.—Revise as shown in the accompanying Table VI.[4]

Standard Specifications for Zinc-Coated (Galvanized) Steel Core Wire (With Coatings Heavier Than Standard Weight) for Aluminum Conductors, Steel Reinforced (ACSR) (B 261 – 58):[2]

Section 4(c).—In Item (*1*) change "Non-Autographic Extensometer" to read "Nonautographic Extensometer." In both Items (*1*) and (*2*) change the first word "If" to read "When."

Section 6(a).—In the second sentence, revise ". . . galvanized ACSR . . ." to read ". . . galvanized steel core wire . . ."

Table VI.—Revise as recommended above for Table VI in Specifications B 245.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Silver-Coated Soft or Annealed Copper Wire (**B 298 – 56 T**),[2] and
Aluminum Wire for Communication Cable (**B 314 – 57 T**).[2]

Tentative Method of Test for:

Stiffness of Bare Soft Square and Rectangular Copper Wire for Magnet Wire Fabrication (**B 279 – 53 T**).[2]

EDITORIAL CHANGE

The committee recommends an editorial change as follows in the Standard Specifications for Bronze Trolley Wire (**B 9 – 55**):[2]

Figure 2.—Due to a typographical error, the wrong cut is shown for Fig. 2. Replace with the cut for Fig. 2 in Specifications B 9 – 53.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Editorial and Records (A. A. Jones, chairman) continued its editorial review of specifications and coordinated the various subcommittee recommendations regarding revisions of specifications.

Subcommittee II on Methods of Test

---

[4] The revised table is not included in this report but appears in the revised specification, see 1959 Supplement to Book of ASTM Standards, Part 2.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

and Sampling Procedures (R. H. Lloyd, chairman) completed preparation of the proposed Tentative Method of Test for Electrical Conductivity by use of Eddy Currents appended to this report.[1] A task group is reconsidering suitable methods of test for determining softness of copper wire.

Subcommittee IV on Conductors of Copper and Copper Alloys (M. V. Yokelson, chairman) completed revisions of Specification B 286 to include silver-coated conductors, and Specification B 174 to clarify length-of-lay requirements and indicate suitable applications, as indicated earlier in this report.

Task groups are cooperating with the U. S. Army Signal Corps Engineering Laboratories in the study of gray-tin transformation; investigating means for measuring wire diameters with a precision greater than 0.1 mil (that is, in hundredths of a mil) and considering degree of significance needed in specifying fine-wire sizes as a preliminary to establishing diameter tolerances for very fine wires; considering a developing need for specifications covering nickel-coated wire and another class (5 per cent) of silver-coated wire; reviewing possible need for revision or expansion of tensile properties requirements for alloy wires (Specifications B 105); and cooperating with Committees B-2 and B-5 to survey the possibility of combining in one test method all of the coating-test requirements now included in Specifications B 33, B 189, and a proposed Committee B-5 specification for coated strip and sheet.

Subcommittee V on Conductors of Ferrous Metals (L. H. Winkler, chairman) prepared the proposed Tentative Specifications for Aluminum-Coated (Aluminized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) appended to this report, and revision of Specifications B 245 and B 261 as noted earlier in the report to include core wires for a revised list of ACSR conductors included in Specification B 232.

Subcommittee VII on Conductors of Light Metals (A. L. Kolb, chairman) completed revision of Specification B 231 to include requirements for testing of wires removed from stranded conductors; and Specification B 232 to include several additional and delete some obsolete sizes of ACSR, and to include requirements for testing of wires removed from stranded conductors. These revisions are listed earlier in the report.

Task groups are investigating the possibility of permitting cold-weld joints in the outer layer of class AA and A aluminum conductors, and reconsidering the inspection requirements in the various aluminum rod and wire specifications.

Joint Task Groups are considering the preparation of standard test report forms for bare electrical conductors and a glossary of terms defining the various terms used in specifications under the jurisdiction of Committee B-1.

This report has been submitted to letter ballot of the committee, which consists of 86 members; 61½ members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. HALLORAN,
Chairman.

A. A. JONES,
Secretary.

REPORT OF COMMITTEE B-2
ON
NON-FERROUS METALS AND ALLOYS*

Committee B-2 on Non-Ferrous Metals and Alloys met on June 25, 1958, in Boston, Mass. Meetings of the Advisory Committee were held on the same date, and also on January 6, 1959, in New York City.

The committee consists of 144 members, of whom 62 are classified as producers, 53 as consumers, and 29 as general interest members.

Appointments during the year have been: D. W. Pettigrew as chairman of Subcommittee IV to replace E. H. Bunce, retired; Warren J. Jenkins as secretary of Subcommittee I; C. J. Snyder as secretary of Subcommittee II; R. I. Jaffee as secretary of Subcommittee VIII; L. L. Wyman to the Thermocouple Committee of Committee E-1; W. C. Bowden, Jr., as representative on Committee E-11; G. O. Hiers on Special Committee on Gaskets and Packing; and J. E. Chard to replace O. B. J. Fraser on Subcommitte IV of Committee E-1.

Subcommittee VIII will sponsor a symposium on the newer metals[1] at the Third Pacific Area National meeting, October 11 to 16, 1959. Dr. R. I. Jaffee is chairman of this symposium.

Drafts have been distributed to the special ASTM committee considering specifications for nickel and nickel alloy fittings; copper-base alloy fittings; and aluminum fittings.

A report was submitted to Committee B-2 by Mr. Smart who is a member of the U. S. delegation of the ISO/TC-26 (Copper) Committee. The directions in which this committee is proceeding were approved by the B-2 Advisory Committee.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On July 25, 1958, the Administrative Committee on Standards, acting for the Society, accepted the following recommendations of Committee B-2:

New Tentative Specifications for:

Nickel-Molybdenum and Nickel-Molybdenum-Chromium Alloy Castings (B 332 - 58 T),
Nickel-Molybdenum Alloy Plate and Sheet (B 333 - 58 T),
Nickel-Molybdenum-Chromium Alloy Plate and Sheet (B 334 - 58 T),
Nickel-Molybdenum Alloy Rod (B 335 - 58 T), and
Nickel-Molybdenum-Chromium Alloy Rod (B 336 - 58 T).

Revision of Tentative Specifications for:

Nickel-Copper Alloy Plate, Sheet, and Strip (B 127 - 58 T),
Nickel Rods and Bars (B 160 - 58 T),
Nickel Seamless Pipe and Tubing (B 161 - 58 T),
Nickel Plate, Sheet, and Strip (B 162 - 58 T),
Seamless Nickel and High Nickel Alloy Condenser, Evaporator, and Heat-Exchanger Tubes (B 163 - 58 T),
Nickel-Copper Alloy Rods and Bars (B 164 - 58 T),
Nickel-Copper Alloy Seamless Pipe and Tubing (B 165 - 58 T),
Nickel-Chromium-Iron Alloy Rods and Bars (B 166 - 58 T),

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

[1] To be issued as separate publication *ASTM STP No. 272.*

Nickel-Chromium-Iron Alloy Seamless Pipe and Tubing (B 167 - 58 T), and
Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (B 168 - 58 T).

The new and revised specifications appear in the 1958 Book of ASTM Standards, Part 2.

On November 13, 1958, the Administrative Committee on Standards accepted the following recommendations:

New Tentative Specifications for:

Seamless and Welded Unalloyed Titanium Pipe (B 337 - 58 T), and
Seamless and Welded Unalloyed Tubing (B 338 - 58 T).

On January 15, 1959, the Administrative Committee on Standards accepted the new Tentative Classification of Pig Tin (B 339 - 59 T).

The new tentatives B 337, B 338, and B 339 will appear in the 1959 Supplement to Book of ASTM Standards, Part 2.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as follows of the Standard Specifications for Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes (**B 170 - 47**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society.

Section 2.—Revise to increase the minimum copper content from "99.92 per cent" to "99.95 per cent."

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specifications for Fire-Refined Casting Copper (**B 72 - 55 T**).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (W. E. Milligan, chairman).—A task group was appointed with C. J. Snyder as chairman to restudy the problem of the inclusion of OFP and OFPT coppers in the Classification of Copper (B 224 - 58).

Subcommittee II on Refined Lead, Tin, Antimony and Bismuth (Sidney Rolle, chairman).—Progress is being made on a specification for nuclear lead. A task group was appointed to revise the scope of Specification B 29 and a report is expected by the Annual Meeting. The subcommittee proposed the Classification of Pig Tin (B 339 - 59 T) which was accepted by the Administrative Committee on Standards, January 15, 1959.

Subcommittee III on Tin-Base and Lead-Base Alloys (G. H. Clamer, chairman).—The title of this subcommittee has been changed from "White Metals" to "Tin-Base and Lead-Base Alloys."

Mr. Herman Smith was appointed chairman of a task group to consider and revise the Specification for White Metal Bearing Alloys (known commercially as "Babbitt Metal") (B 23 - 49). Also, Specifications B 32 and B 284 are being revised.

A Symposium on Solder is being considered for 1961.

Subcommittee IV on Refined Zinc and Wrought Zinc (D. W. Pettigrew, chairman).—D. W. Pettigrew was appointed as chairman of this subcommittee to replace E. H. Bunce, retired. Revisions are being made in the Specification for Slab Zinc (Spelter) (B 6 - 58).

Subcommittee VII on Refined Nickel

---

[2] 1958 Book of ASTM Standards, Part 2.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

and Cobalt, High Nickel Alloys and High Cobalt Alloys, Cast and Wrought (V. W. J. Franceschini, chairman).—All of the specifications under the jurisdiction of this subcommittee, with the exception of Specifications B 39, were revised and have been accepted for inclusion in the 1958 Book of ASTM Standards. New specifications B 332, B 333, B 334, B 335, and B 336 were also accepted by the Administrative Committee on Standards.

A joint coordinating committee of members of Committees A-1, A-10, B-2, B-4, and F-1 was appointed to consider specifications for cast and wrought forms of nickel-chromium, nickel-chromium-iron, nickel-copper, and other nickel-base alloys (over 50 per cent nickel).

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman).—The nine task groups set up to study the less common metals, columbium, molybdenum, zirconium, lithium, beryllium, tantalum, tungsten, thorium, and uranium, have all been active and work is continuing to develop specifications for these metals where needed. Tentative specifications for molybdenum and molybdenum alloy strip, sheet, foil, and plate have been drafted; also drafts have been prepared for tentative specifications for zirconium and beryllium for nuclear purposes.

A member of the subcommittee will be appointed to represent B-2 on Committee E-2 to aid in the collection and promulgation of analytical methods for the newer metals.

Subcommittee IX on Titanium and Titanium Alloys (T. W. Lippert, chairman).—New specifications for titanium pipe and tubing (B 337 and B 338) have been accepted by the Administrative Committee on Standards. Work is continuing on a specification for titanium and titanium alloy bar.

This report has been submitted to letter ballot of the committee, which consists of 144 members; 105 members returned their ballots, of whom 101 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

BRUCE W. GONSER,
Chairman.

A. M. BOUNDS,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Titanium and Titanium Alloy Bars and Billets (B 348 – 59 T).

Revision and Reversion to Tentative of Standard Specifications for:

White Metal Bearing Alloy (Known Commercially as "Babbitt Metal") (B 23 – 49).

The new Tentative Specification B 348 – 59 T was accepted by the Standards Committee on September 15, 1959, and the revision of Standard Specification B 23 – 49 was accepted on November 6, 1959. The new and revised specifications are available as separate reprints.

REPORT OF COMMITTEE B-3
ON
CORROSION OF NON-FERROUS METALS AND ALLOYS*

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held two meetings during the past year: in Boston, Mass., on June 25, 1958, and in Pittsburgh, Pa., on February 2, 1959.

The committee consists of 101 members, of whom 84 are voting members; 33 are classified as producers, 30 as consumers, and 21 as general interest members.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

Salt Spray (Fog) Testing (**B 117 - 57 T**),
Total Immersion Corrosion Test of Non-Ferrous Metals (**B 185 - 43 T**), and
Alternate Immersion Corrosion Test of Non-Ferrous Metals (**B 192 - 44 T**).

EDITORIAL CHANGE

The committee recommends editorial changes as follows in the Tentative Method of Acetic Acid-Salt Spray (Fog) Testing (**B 287 - 57 T**):[1]

Section 6.—In the third sentence delete the words "approximately 1 per cent of" so that the sentence shall read: "The pH of this solution shall be adjusted to the range 3.1 to 3.3 by the addition of acetic acid."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Spray Test (C. O. Durbin, chairman).—A task group is continuing studies to determine the limits to be set for heavy metals in salt used in salt spray tests.

Work done by the task group indicates that copper salts may be useful in effectively accelerating the acetic acid - salt spray test.

Subcommittee VI on Atmospheric Corrosion (H. R. Copson, chairman).—All specimens for a new series of atmospheric tests initiated in 1957 have been placed on exposure. A report giving composition of the metals and their initial tensile properties is appended to this report.[3]

Subcommittee VII on Weather (C. P. Larrabee, chairman).—A report on the calibration of the corrosivity of 19 atmospheric test sites, using steel and zinc specimens exposed for various periods of time, is appended to this report.[4]

In accordance with a request from the Advisory Committee on Corrosion, new calibration tests of 9 ASTM test sites and 20 test sites under private jurisdiction will be initiated in 1960.

A task group is working on the possible

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] 1958 Book of ASTM Standards, Part 3.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[3] See p. 176.

[4] See p. 183.

correlation of atmospheric contaminants and weight loss of steel and zinc at certain test sites.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (G. V. Kingsley, chairman).—The panel-type galvanic couples of magnesium with other metals were exposed on the following dates:

State College, Pa. (rural)	September 25, 1958
Newark, N. J. (New York City Area) (industrial)	October 10, 1958
Kure Beach, N. C. (marine):	
80-ft site	December 6, 1958
800-ft site	December 6, 1958

A task group has been appointed to look into the feasibility of the subcommittee undertaking a study of the corrosion of tube materials used in boiler feed-water heaters.

The subcommittee was interested in having submitted to it a report by A. Gallaccio and I. Cornet, on Marine Atmosphere Exposure of Galvanic Couples Involving Magnesium, published as a separate technical publication *ASTM STP No. 255*).

This report has been submitted to letter ballot of the committee, which consists of 84 voting members; 63 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

A. W. TRACY,
Secretary.

APPENDIX I

REPORT OF SUBCOMMITTEE VI ON ATMOSPHERIC CORROSION

1957 TEST PROGRAM

One of the earliest committees of the Society to recognize the necessity for long-term atmospheric, as opposed to accelerated, corrosion tests was Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys. In 1931 this group exposed some 9000 panels at nine different locations. The 24 metals included alloys of zinc, nickel, copper, lead, tin, and aluminum. The 10-yr results of these atmospheric corrosion tests were presented in 1946 and published by the Society as the Symposium on Atmospheric Corrosion of Non-Ferrous Metals.[1] A similar Symposium was held in 1955, published under the same title,[2] that summarized the results of these exposures after 20 yr, the predetermined test duration.

Recognizing the value to industry of these earlier tests and cognizant of the fact that newer alloys with supposedly vastly improved corrosion resistance had been developed, Committee B-3, in 1956, initiated a new series of atmospheric corrosion tests, under a task group consisting of J. S. Pettibone, chairman, W. H. Ailor, A. W. Tracy, and L. Whitby. A questionnaire was sent to all members of the committee to determine the type of alloys to be included in the program, sites at which specimens should be exposed, and information as to how corrosion damage should be evaluated.

MATERIALS

The 1957 program contains 77 alloys of 9 basis metals including some of the newer commercial metals such as titanium, tantalum, and molybdenum. The number of alloys in each category is as follows:

Aluminum	34
Copper	19
Lead	2
Magnesium	5
Molybdenum	1
Nickel	5
Tantalum	1
Titanium	8
Zinc	2

TEST PROGRAM

Atmospheric Exposure:

Test Site Locations.—Site A: Kure Beach (80-ft site), N. C. (East Coast Marine); Site B: New York Area (Newark, N. J.) (Industrial); Site C: Point Reyes, Calif. (West Coast Marine); and Site D: State College (University Park), Pa. (Rural).

Test Periods.—Initial tests (zero point), atmospheric exposures for 2, 7, and 20 yr (triplicate panels), storage for 2, 7, and 20 yr (duplicate panels), plus a one year period for aluminum alloys.

Test Panels.—8 by 4 by 0.05 in. (see Fig. 1), with some variations in thickness, were exposed at 30 deg from the horizontal. All panels of each alloy are from one heat with the 8-in. dimension parallel to the direction of rolling.

---

[1] Issued as a separate publication *ASTM STP No. 67* (1946).

[2] Issued as a separate publication, *ASTM STP No. 175* (1955).

Identification.—Panels were marked with ¼-in. metal stencils. Identification was made as shown in Table I and II and Fig. 1. Table II shows the number arbitrarily assigned to each alloy in the program with its commercial designation. The six 7-yr specimens (2 specimens from each of 3 panels) of any alloy at Kure Beach may be identified by numbers from 25 to 30, by the location, *A*, and alloy designation as shown in Table I. Marking was in accordance with Fig. 1: alloy designation at top, followed by test site in center, and then specimen number.

Laboratory Tests:

Chemical Analyses were made on all 77 alloys (see Table II).

Thickness Measurements were made to the nearest 0.001 in. on the specific locations shown in Fig. 1.

Length and Width measurements were made to the nearest 0.01 in.

Weighing of the aluminum and magnesium alloys was to the nearest 0.001 g. All other alloys were weighed to the nearest 0.01 g.

Initial Tension Tests were made on six specimens, three each from two panels (see Table III).

Evaluation of Corrosion Damage:

Weighing.—All panels will be cleaned and weighed to the nearest 0.01 g, except the aluminum and magnesium panels which will be weighed to the nearest 0.001 g.

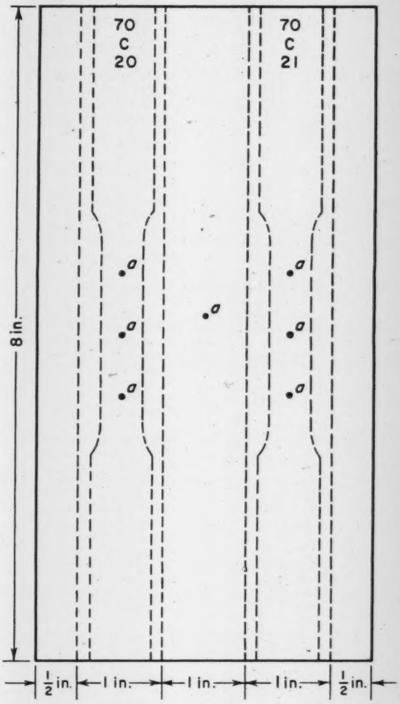


FIG. 1.—Exposure Panel, Indicating Location of Tension Test Bars and Marking Code.

The code indicates: Alloy number at top, test site location letter, and specimen number. Points designated *a* are the points at which thickness measurements are made.

Tension Tests will be made on two specimens cut from each of three exposed panels after cleaning and weighing.

Pit Depth Determinations will be made on the center section of each panel after removal of tension specimens.

TABLE I.—CODE FOR IDENTIFICATION OF ATMOSPHERIC EXPOSURE LOCATION.

Location of Test Site	Code	1 Year	2 Year	7 Year	20 Year
Kure Beach, N. C.	A	73 to 78	1 to 6	25 to 30	49 to 54
New York Area (Newark, N. J.)	B	79 to 84	7 to 12	31 to 36	55 to 60
Point Reyes, Calif.	C	85 to 90	13 to 18	37 to 42	61 to 66
State College, Pa.	D	91 to 96	19 to 24	43 to 48	67 to 72

TABLE II.—CHEMICAL ANALYSES.

ALUMINUM ALLOYS.

Alloy	Commercial Designation	Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Nickel, per cent
No. 10...	1188-H14..........	Remainder	0.000	0.044	0.036	0.00	0.000	0.00	0.00	0.004	...
No. 11...	1135-H14..........	Remainder	0.12	0.31	0.06	0.00	0.00	0.00	0.00	0.02	...
No. 12...	3004-H34..........	Remainder	0.14	0.48	0.19	1.08	1.06	0.04	0.01	0.02	...
No 13...	4043-H14..........	Remainder	0.01	0.27	5.67	0.01	0.00	0.00	0.00	0.01	...
No. 14...	5454-0............	Remainder	0.06	0.24	0.14	0.75	2.84	0.02	0.09	0.01	...
No. 15...	5454-H34..........	Remainder	0.05	0.23	0.13	0.74	2.82	0.02	0.09	0.01	...
No. 16...	Clad 5155-H34										
	Core..........	Remainder	0.07	0.29	0.14	0.48	4.42	0.04	0.12	0.04	...
	Cladding........	Remainder	0.01	0.28	0.09	0.02	0.01	1.24	0.00	0.01	...
No. 17...	5456-0............	Remainder	0.05	0.27	0.16	0.77	5.32	0.02	0.12	0.05	...
No. 18...	1100-H14..........	Remainder	0.15	0.58	0.10	0.02	0.00	0.00	0.00	0.01	...
No 19...	2014-T4...........	Remainder	4.74	0.37	0.86	0.72	0.46	0.20	0.05	0.02	...
No. 20...	Clad 2014-T6										
	Core...........	Remainder	4.74	0.39	1.13	0.83	0.53	0.15	0.04	0.01	...
	Cladding........	...	...	...	0.35 to 1.0	0.8 max	0.8 to 1.5	...	...	...	...
No. 21...	Clad 2024-T3										
	Core..........	Remainder	4.55	0.26	0.16	0.64	1.63	0.08	0.00	0.01	...
	Cladding.......	99.3	...	...	...	...	...	...	...	...	...
No. 22...	3003-H14..........	Remainder	0.13	0.59	0.24	1.01	0.01	0.07	0.00	0.01	...
No. 23...	5005-H34..........	Remainder	0.11	0.58	0.20	0.06	0.79	0.08	0.03	0.01	...
No. 24...	5050-H34..........	Remainder	0.10	0.57	0.16	0.05	1.40	0.04	0.03	0.02	...
No. 25...	5052-H34..........	Remainder	0.07	0.23	0.07	0.00	2.40	0.08	0.21	0.00	...
No. 26...	5083-0	Remainder	0.04	0.22	0.10	0.64	4.63	0.06	0.12	0.03	...
No. 27...	5083-H34..........	Remainder	0.04	0.26	0.11	0.50	3.96	0.02	0.11	0.01	...
No. 28...	7075-T6...........	Remainder	1.70	0.25	0.13	0.09	2.60	5.81	0.20	0.03	...
No. 29...	Clad 7075-T6										
	Core...........	Remainder	1.62	0.27	0.14	0.09	2.73	5.60	0.18	0.04	...
	Cladding.......	Remainder	...	...	...	...	...	1.0	...	...	...
No. 30...	2024-T3...........	Remainder	4.60	0.35	0.22	0.56	1.49	[a]	[a]	[a]	[a]
No. 31...	2024-T36..........	Remainder	4.54	0.39	0.22	0.58	1.43	[a]	[a]	[a]	[a]
No. 32...	2024-T81..........	Remainder	4.49	0.35	0.20	0.55	1.44	[a]	[a]	[a]	[a]
No. 33...	2024-T86..........	Remainder	4.67	0.31	0.24	0.56	1.44	[a]	[a]	[a]	[a]
No. 34...	Clad										
	Core, 3003-H14..	Remainder	0.14	0.54	0.20	1.10	0.02	0.09	...	<0.01	...
	Cladding, 7072...	Remainder	0.03	0.26	0.11	<0.01	0.02	1.00	...	...	...
No. 35...	5154-H34..........	Remainder	[a]	0.24	0.11	[a]	3.40	[a]	0.20	[a]	[a]
No. 36...	5357-H34..........	Remainder	[a]	...	...	0.31	1.01	...	...	...	...
No. 37...	6061-T4...........	Remainder	0.33	0.48	0.70	[a]	0.85	[a]	0.20	[a]	[a]
No. 38...	6061-T6...........	Remainder	0.33	0.41	0.68	[a]	0.90	[a]	0.20	[a]	[a]
No. 39...	Clad										
	Core, 6061-T6...	Remainder	0.32	0.42	0.63	...	0.84	...	0.25	...	...
	Cladding, 7072..	Remainder	0.03	0.22	0.11	<0.01	0.04	1.14	...	...	...
No. 84...	5083-H34..........	Remainder	0.04	0.22	0.10	0.64	4.63	0.06	0.12	0.03	...
No. 85...	X7079-T6..........	Remainder	0.66	0.23	0.13	0.19	3.38	4.48	0.18	0.07	...
No. 86...	Clad X7079-T6										
	Core...........	Remainder	0.65	0.23	0.12	0.19	3.34	4.45	0.18	0.07	...
	Cladding........	Remainder	0.01	0.28	0.09	0.02	0.01	1.24	0.00	0.01	...
No. 87...	Super purity.......	Remainder	0.0015	0.0035	0.0025	0.0005	...	...	...	...	...

COPPER ALLOYS

Alloy	Commercial Designation	Copper, per cent	Zinc, per cent	Tin, per cent	Phosphorus, per cent	Lead, per cent	Iron, per cent	Nickel, per cent	Manganese, per cent	Aluminum, per cent	Silicon, per cent	Beryllium, per cent	Other, per cent
No. 65...	1.25 Sn Phosphor bronze..........	98.71	<0.10	1.18	0.11	...	...	...	...	...	...	...	...
No. 66...	90-10 Cupro-nickel	88.23	0.10	...	...	<0.02	1.23	10.12[b]	0.32	...	...	...	...
No. 67...	5 Sn Phosphor bronze..........	[b]	0.04	4.08	0.16	0.001	0.005	0.00	...	0.00	0.00	...	...
No. 68...	Tin brass..........	88.37	...	1.83	0.10	0.003	0.015	0.00	...	0.00	0.01	...	...
No. 69...	2 Si-7 Al bronze...	91.65	0.02	0.00	...	0.001	0.02	0.00	...	6.40	1.85	...	0.06, arsenic
No. 70...	Red brass.........	84.71	15.25[b]	<0.01	...	<0.05	0.02	0.02	...	...	...	...	...
No. 71...	Cartridge brass....	70.60	29.38[b]	<0.01	...	<0.05	0.014	<0.01	...	...	...	...	...
No. 72...	10 per cent Nickel silver..........	64.58	24.46[b]	...	...	0.01	0.01	10.53	0.41	...	...	...	...
No. 73...	18 per cent Nickel silver..........	65.14	15.87[b]	...	...	0.01	0.02	18.56	0.40	...	...	...	...
No. 74...	Tough-pitch copper............	99.940[c]	...	...	...	...	...	...	...	...	...	...	0.042, oxygen 0.003, sulfur
No. 75...	No. 30............	97.99	...	...	...	...	0.03	1.85	0.02	...	...	...	...
No. 76...	No. 60............	93.73	...	...	...	...	0.01	6.34	<0.01	...	...	...	...
No. 77...	No. 95............	90.35	...	...	...	...	0.18	9.16	0.27	...	...	...	...
No. 78...	No. 180...........	77.18	...	...	...	...	0.04	22.76	<0.01	...	...	...	...
No. 79...	Advance...........	55.38	...	...	...	...	0.30	42.75	1.67	...	...	...	...
No. 80...	No. 212...........	82.62	...	...	...	...	0.20	4.13	12.83	...	...	...	...
No. 81...	Beraloy A.........	[b]	...	...	...	...	0.11	...	...	0.11	0.10	1.89	0.26, cobalt
No. 82...	Beraloy D.........	[b]	...	...	...	...	0.07	...	...	0.15	0.08	1.76	0.27, cobalt
No. 83...	Beraloy C.........	[b]	...	...	...	...	0.08	...	...	0.05	0.05	0.54	2.56, cobalt

TABLE II.—CHEMICAL ANALYSES—*Continued.*

Lead Alloys

Alloy	Commercial Designation	Lead, per cent	Silver, per cent	Copper, per cent	Bismuth, per cent	Tin, per cent	Arsenic, per cent	Nickel, per cent	Zinc, per cent	Cadmium, per cent	Antimony, per cent	Iron, per cent
No. 40....	Chemical lead	[b]	0.0071	0.06	<0.005	<0.0001	<0.0008	0.0038	<0.0005	0.0023	<0.001	<0.0005
No. 41....	6 per cent Antimonial lead.....	[b]	0.0077	0.053	<0.001	<0.01	<0.01	0.0019	<0.001	0.0016	5.62	...

Magnesium Alloys

Alloy	Commercial Designation	Magnesium, per cent	Aluminum, per cent	Calcium, per cent	Copper, per cent	Iron, per cent	Manganese, per cent	Nickel, per cent	Lead, per cent	Silicon, per cent	Zinc, per cent	Thorium, per cent	Zirconium, per cent	Rare Earths, per cent
No. 42....	AZ31B-H24......	[b]	3.0	0.016	0.007	0.001	0.54	<0.001	<0.001	<0.01	1.00	...	...	...
No. 43....	HK31A-H24.....	[b]	<0.03	<0.01	0.006	0.001	0.048	<0.001	<0.001	<0.01	<0.02	3.23	0.66	...
No. 44....	HM21XA-T8.....	[b]	<0.03	<0.01	0.005	0.001	0.50	<0.001	<0.001	<0.01	<0.02	2.15	...	...
No. 45....	ZE10XA-H24....	[b]	<0.03	<0.01	0.007	0.021	0.071	<0.001	<0.001	<0.01	1.13	...	...	0.22
No. 46....	ZH11X1-H24.....	[b]	<0.03	<0.01	0.003	0.003	0.044	<0.001	<0.001	<0.01	1.00	0.95	0.61	0.51

Molybdenum Alloys

Alloy	Commercial Designation	Molybdenum, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Copper, per cent	Nickel, per cent	Magnesium, per cent
No. 47....	Molybdenum...	[b]	0.005 to 0.01	0.01 to 0.1	0.001 to 0.005	<0.0001	0.01 to 0.1	0.001 to 0.01

Nickel Alloys

Alloy	Commercial Designation	Nickel,[d] per cent	Chromium, per cent	Carbon, per cent	Manganese, per cent	Iron, per cent	Sulfur, per cent	Silicon, per cent	Copper, per cent	Aluminum, per cent	Titanium, per cent	Magnesium, per cent	Boron, per cent	Molybdenum, per cent	Columbium, per cent
No. 48...	"A" Nickel...	99.28	...	0.03	0.28	0.30	0.008	0.03	0.05	...	0.018	0.033	...	...	...
No. 49...	Incoloy.......	32.80	21.08	0.06	0.83	44.58	0.007	0.38	0.24	0.42	0.48	...	...	...	...
No. 50...	Inconel........	75.82	16.04	0.03	0.19	7.61	0.007	0.18	0.10	0.09	0.21	0.026	0.008	...	...
No. 51...	Monel.........	66.77	...	0.10	0.86	1.34	0.010	0.14	30.76	0.007	0.009	0.051	...	...	...
No. 52...	Ni-O-Nel......	41.59	22.16	0.03	0.56	29.03	0.009	0.34	1.77	0.09	0.40	...	...	3.05	0.95

Tantalum Alloys

		Tantalum, per cent	Aluminum, per cent	Silicon, per cent	Titanium, per cent	Iron, per cent	Columbium, per cent	Nitrogen, per cent	Hydrogen, per cent	Oxygen, per cent	Carbon, per cent
No. 64.......	Tantalum.....	[b]	0.002	0.012	0.003	0.008	0.01	0.015	<0.001	0.011	0.004

Titanium Alloys

Alloy	Commercial Designation	Titanium, per cent	Carbon, per cent	Nitrogen, per cent	Iron, per cent	Aluminum, per cent	Vanadium, per cent	Chromium, per cent	Manganese, per cent	Columbium, per cent	Molybdenum, per cent	Tantalum, per cent	Oxygen, per cent	Hydrogen, per cent	Tin, per cent
No. 53...	6 Al-4 V titanium....	[b]	0.04	0.010	0.22	6.37	4.12	...	...	...	...	...	0.102	0.0047	...
No. 54...	8 Mn titanium......	[b]	0.02	0.023	0.18	...	...	...	8.30	...	...	...	0.160	0.0048	...
No. 55...	2.5 Al-16 V titanium..	[b]	0.03	0.016	0.20	2.82	16.40	...	...	...	...	...	0.114	0.0085	...
No. 56...	821..........	[b]	0.02	0.013	0.22	7.96	...	...	...	2.33	...	1.01	0.092	0.0095	...
No. 57...	75A.........	[b]	0.013	0.009	0.08	...	...	...	...	...	...	...	...	0.006	...
No. 58...	140A........	[b]	0.012	0.014	1.75	...	...	2.07	...	...	1.87	...	...	0.008	...
No. 59...	5 Al-2.5 Sn titanium...	[b]	0.013	0.012	0.09	5.8	...	...	...	...	...	...	...	0.006	2.4
No. 60...	4 Al-3 Mo-1 V titanium...	[b]	0.016	0.013	0.09	4.36	0.90	...	...	...	2.90	...	...	0.001	...

TABLE II.—CHEMICAL ANALYSES—*Concluded.*

ZINC ALLOYS

Alloy	Commercial Designation	Zinc, per cent	Lead, per cent	Iron, per cent	Cadmium, per cent	Copper, per cent
No. 61	High grade zinc	[b]	0.058	0.007	0.009	...
No. 62	1 Cu Zinc	[b]	0.093	0.012	0.004	0.79

[a] Less than the allowable maximum.
[b] By difference.
[c] Copper plus silver.
[d] Contains small amount of cobalt.

Storage of Panels:

The storage panels were wrapped in moisture-proof paper, then dipped in microcrystalline wax in order to maintain similar conditions of humidity and temperature. Each alloy was divided into three lots (four for aluminum) and identified by marking the inner wrapping so that identification could be made without the necessity of breaking the transparent wax seal. The ASTM Headquarters is storing the storage panels for this program.

ACQUISITION OF MATERIALS

After preliminary approval of this program by the main committee, the task group approached various producers for panels of the alloys desired in the program. The companies which furnished not only material and laboratory services but man-power and equipment necessary to perform the preliminary tests as well are:

Aluminum Company of America
Aluminum Development Assn.
The American Brass Co.
Bridgeport Brass Co.
Chase Brass and Copper Co.
The Dow Chemical Co.
Driver-Harris Co.
The International Nickel Co., Inc.
Kaiser Aluminum and Chemical Corp.
Kawecki Chemical Co.
Mallory Sharon Metals Corp.
National Lead Co.
New Haven Copper Co.
The New Jersey Zinc Co.
Reynolds Metals Co.
Scovill Manufacturing Co.
Titanium Metals Corporation of America
Westinghouse Electric Corp.
Wilbur B. Driver Co.

EXPOSURE OF SPECIMENS

Each alloy was exposed in triplicate for each exposure period at all four sites with one exception: sufficient tantalum was available for exposure only at Kure Beach, N. C., and Newark, N. J. The total number of all panels exposed was 3162. This is considerably less than the 9000 exposed in the 1931 tests. However, it was felt that the nine sites used in the earlier tests could be judiciously reduced to four. In addition, the number of exposure periods is less than used in the previous tests. The actual exposure test dates are shown in Table IV.

It is a credit to the many people involved that the actual exposure of specimens was made less than 2 yr after Committee B-3 authorized the task group to proceed with this new program. The committee is fully cognizant of the time, labor, and thought expended by the many contributors, and it is hoped that the resulting data will be as valuable as that obtained from the earlier tests.

Respectfully submitted on behalf of the subcommittee,

H. R. COPSON,
Chairman.

TABLE III.—INITIAL TENSION TEST DATA.

Alloy	Average Thickness, in.	Width, in.	Area, sq in.	Load, lb	Tensile Strength, psi	Elongation in 2 in., per cent
No. 10	0.052	0.499	0.026	307	11 930	12.3
No. 11	0.051	0.501	0.026	431	16 870	10.2
No. 12	0.051	0.501	0.026	940	36 470	8.8
No. 13	0.051	0.502	0.026	595	23 030	9.9
No. 14	0.052	0.500	0.026	963	37 180	21.1
No. 15	0.052	0.497	0.026	1184	46 000	8.7
No. 16	0.053	0.498	0.026	1382	52 580	9.8
No. 17	0.054	0.499	0.027	1342	50 270	22.3
No. 18	0.050	0.500	0.025	462	18 620	6.7
No. 19	0.050	0.499	0.025	1707	69 120	19.8
No. 20	0.050	0.500	0.025	1739	69 320	9.2
No. 21	0.050	0.500	0.025	1647	65 980	17.6
No. 22	0.050	0.500	0.025	553	22 020	7.0
No. 23	0.050	0.499	0.025	554	22 100	6.7
No. 24	0.052	0.498	0.026	710	27 650	6.2
No. 25	0.051	0.500	0.025	906	35 620	9.2
No. 26	0.049	0.499	0.025	1110	44 880	20.1
No. 27	0.050	0.500	0.025	1221	48 470	9.7
No. 28	0.049	0.500	0.024	2005	82 000	9.8
No. 29	0.049	0.501	0.025	1901	76 780	10.8
No. 30	0.054	0.496	0.027	1852	69 400	19.8
No. 31	0.053	0.496	0.026	1883	72 200	14.8
No. 32	0.050	0.501	0.025	1781	71 000	6.2
No. 33	0.051	0.507	0.026	1993	77 100	5.5
No. 34	0.051	0.502	0.026	545	21 200	6.0
No. 35	0.050	0.509	0.026	1098	43 100	9.8
No. 36	0.052	0.509	0.026	654	24 700	7.4
No. 37	0.050	0.512	0.026	1022	39 900	20.9
No. 38	0.051	0.508	0.026	1209	46 000	11.0
No. 39	0.050	0.515	0.026	1154	44 800	13.0
No. 40	0.053	0.493	0.026	60.5	2 315	47.1
No. 41	0.050	0.492	0.024	96.7	3 925	58.1
No. 42	0.063	0.502	0.032	1285	40 600	20.3
No. 43	0.061	0.504	0.031	1098	35 670	9.3
No. 44	0.063	0.503	0.031	1093	34 720	5.9
No. 45	0.063	0.502	0.032	1241	38 950	13.7
No. 46	0.058	0.504	0.030	1112	37 700	17.0
No. 47	0.050	0.490	0.025	2417	95 570	15.7
No. 48	0.049	0.496	0.024	1491	62 150	42.5
No. 49	0.050	0.496	0.024	2113	88 040	45.0
No. 50	0.051	0.497	0.025	2337	93 580	44.5
No. 51	0.050	0.497	0.025	1838	74 560	41.0
No. 52	0.050	0.499	0.025	2195	87 810	49.0
No. 53	0.064	0.504	0.0325	4276	131 600	9.6
No. 54	0.055	0.506	0.0275	4043	147 000	17.6
No. 55	0.043	0.503	0.022	3815	173 300	4.9
No. 56	0.036	0.502	0.0185	2301	124 200	17.2
No. 57	0.0401	0.496	0.0199	1831	91 900	27.0
No. 58	0.0405	0.496	0.0201	2643	132 000	19.6
No. 59	0.0463	0.495	0.0228	2800	123 000	18.3
No. 60	0.0435	0.496	0.0216	2820	131 000	14.0
No. 61	0.0484	0.498	0.0241	463	19 200	63.0
No. 62	0.0486	0.497	0.0241	566	23 500	52.8
No. 64	...	...	0.0111	835	75 280	2.4
No. 65	0.050	0.496	0.025	1036	41 850	48.4
No. 66	0.049	0.499	0.025	1211	49 370	34.4
No. 67	0.0485	0.5059	0.0246	1207	49 180	52
No. 68	0.0491	0.5051	0.0246	1143	46 100	54
No. 69	0.0518	0.5039	0.0248	2007	76 980	51
No. 70	0.052	0.502	0.026	1100	42 000	43.3
No. 71	0.052	0.499	0.026	1057	48 400	57.8
No. 72	0.049	0.505	0.025	1381	56 000	47.0
No. 73	0.048	0.505	0.024	1373	56 330	40.8

TABLE III.—INITIAL TENSION TEST DATA—*Concluded.*

Alloy	Average Thickness, in.	Width, in.	Area, sq in.	Load, lb	Tensile Strength, psi	Elongation in 2 in., per cent
No. 74	0.049	0.500	0.025	855	34 580	45.5
No. 75	0.051	0.509	0.026	801	30 700	37.6
No. 76	0.050	0.496	0.025	826	33 040	29.9
No. 77	0.052	0.562	0.029	1013	34 930	35.7
No. 78	0.049	0.499	0.024	1160	48 330	32.6
No. 79	0.054	0.495	0.027	1535	56 850	38.6
No. 80	0.052	0.508	0.026	1476	56 770	34.8
No. 81	0.050	0.499	0.025	1815	72 800	39.8
No. 82	0.050	0.494	0.025	1730	69 100	40.8
No. 83	0.050	0.496	0.025	1260	50 600	34.0
No. 84	0.052	0.500	0.026	1408	53 680	11.2
No. 85	0.052	0.499	0.026	2024	77 900	12.2
No. 86	0.051	0.499	0.025	1852	72 780	11.5
No. 87	0.049	0.506	0.025	406	16 400	6.0

TABLE IV.—EXPOSURE DATES.

Kure Beach (80-ft site), N. C. (Site A)	Nov. 22, 1958
Newark, N. J. (Site B)	Oct. 31, 1958
Point Reyes, Calif. (Site C)	
Alloys 10 to 36	May 24, 1958
Alloys 37 to 74 (except No. 63) and 81 to 86	June 11, 1958
Alloys 75 to 80	May 19, 1958
Alloy 87	Jan. 22, 1959
State College (University Park), Pa. (Site D)	Nov. 6, 1958

APPENDIX II

REPORT OF SUBGROUP[1] OF SUBCOMMITTEE VII, ON CORROSIVENESS OF VARIOUS ATMOSPHERIC TEST SITES AS MEASURED BY SPECIMENS OF STEEL AND ZINC

During the February, 1948, meeting of Subcommittee VII on Weather, of ASTM Committee B-3 on Corrosion of Non-Ferrous Metals, the effects of periodic variation in weather conditions on atmospheric corrosion were discussed. To determine the magnitude of such variations C. P. Larrabee and O. B. Ellis were appointed as a task group to make periodic exposures of samples of mild steel and standard rolling grade zinc at several atmospheric test sites. The first exposures were made in April, 1948. Annual exposures and removals were made as planned from April, 1948, through April, 1956. Data resulting from exposure of specimens for four successive periods of 1 yr each were reported at the 1953 Annual Meeting.[2] This is a final report of the investigation as originally planned.

PLAN OF TEST

The test program was limited to two materials: cold-rolled steel and sheet zinc. Four specimens, 4 by 6 in., of each material were exposed for each removal period at each test site. The steel specimens were identified by notches punched in the edges, and the zinc specimens by drilled holes. The identification code designated the time of exposure and removal, and the location of the specimens.

The steel specimens were pickled and weighed before exposure. After exposure they were cleaned in molten sodium hydroxide - sodium hydride and reweighed. The zinc specimens were degreased and weighed before exposure. After removal from the test, they were cleaned in dilute ammonium hydroxide and reweighed.

The specimens were mounted on open frames between porcelain insulators. The frames were at an angle of 30 deg from the horizontal, facing south.

Several samples of steel and of zinc, each consisting of four 4 by 6-in. specimens, were exposed according to the schedule shown in Table I. From this table it is evident that cumulative data for exposure periods greater than 1 yr were obtained.

PRESENTATION OF DATA

All of the available data on the losses of weight of steel specimens after various periods are presented in Tables II to IX and of zinc specimens in Tables X to XIX. From these tables it is evident that there are annual variations in weight losses at all locations and that the several locations showed differing severity of corrosive attack. Attention is called to the fact that at one location (New York,

---

[1] C. P. Larrabee, Chief Research Engineer—Corrosion Prevention, Applied Research Laboratory, United States Steel Corp., Monroeville, Pa., and O. B. Ellis, Senior Research Engineer, Armco Steel Corp., Middletown, Ohio.

[2] "Progress Report of a Subgroup of Subcommittee VII, On Corrosiveness of Various Atmospheric Test Sites as Measured by Specimens of Steel and Zinc," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 194 (1953).

TABLE I.—SCHEDULE FOR EXPOSURE AND REMOVAL OF ATMOSPHERIC CALIBRATION TESTS.

Year Exposed	Year Removed								Number of Years of Exposure
	1949	1950	1951	1952	1953	1954	1955	1956	
1948	×	×	...	×	...	...	...	×	1, 2, 4, 8
1949	...	×	×	...	×	...	...	×	1, 2, 4, 7
1950	...	...	×	×	...	×	...	×	1, 2, 4, 6
1951	...	...	...	×	×	...	×	×	1, 2, 4, 5
1952	...	...	...	...	×	×	...	×	1, 2, 4
1953	...	...	...	...	...	×	×	×	1, 2, 3
1954	...	...	...	...	...	...	×	×	1, 2
1955	...	...	...	...	...	...	...	×	1

TABLE II.—STEEL EXPOSURES AT STATE COLLEGE, PA., RURAL ATMOSPHERE.

Loss of Weight, g, 4 by 6-in. Steel Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4yr		Exposed to 1956	
	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]
1948	6.9	0.07	11.5	0.14	18.2	0.11	(8 yr) 25.7	0.16
1949	6.9	0.07	13.6	0.10	16.9	0.18	(7 yr) 22.6	0.15
1950	8.8	0.15	11.6	0.05	18.9	0.19	(6 yr) 20.7	0.11
1951	6.1	0.05	10.1	0.07	16.6	0.16	(5 yr) 17.4	0.07
1952	5.9	0.13	11.3	0.11	14.3	0.44	...	...
1953	6.7	0.05	10.5	0.07	...	...	(3 yr) 12.4	0.05
1954	7.1	0.17	10.1	0.05	...	...	...	...
1955	6.1	0.14	...	...	...	...	...	...
Average	6.8	0.86[b]	11.2	1.13[b]	17.0	1.58[b]	...	...

[a] Standard Deviation of 4 specimens.
[b] Standard Deviation of the yearly losses.

TABLE III.—STEEL EXPOSURES AT NEW YORK, N. Y., INDUSTRIAL ATMOSPHERE, SPRING INSTALLATION.

Loss of Weight, g, 4 by 6-in. Steel Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]
1949	19.5	0.17	33.0	0.33	39.3	1.49	(7 yr) 48.4	3.45
1950	35.3	0.22	41.5	2.07	51.4	1.42	(6 yr) 60.1	1.64
1951	17.6	0.20	23.6	0.24	29.3	0.69	(5 yr) 33.6	0.60
1952	19.3	0.37	27.3	1.00	33.6	0.70	...	...
1953	16.8	0.35	21.0	0.33	...	...	(3 yr) 26.3	1.03
1954	13.9	0.28	25.5	2.07	...	...	...	...
1955	25.6	0.68	...	...	...	...	...	...
Average	21.1	6.46[b]	28.7	6.83[b]	38.4	9.57[b]	...	...

[a] Standard Deviation of 4 specimens.
[b] Standard Deviation of the yearly losses.

N. Y.) exposures were made in both the spring (Tables III and XI) and the fall (Tables VIII and XVI). Tables IX, XVII, XVIII, and XIX contain the data from 14 sites where for various reasons the data are not complete. Many of these data are expressed graphically in Figs. 1 to 9.

The exposure work in Canada was done with the cooperation of the National Research Council of Canada at test sites under its jurisdiction (sites 1, 2, 3, 6, 8, 9, 10, and 17). These sites have been described by the National Research Council.[3] Sites 5, 12, 13, 14, 15, and 19 were official ASTM test sites. However, since the start of this program, site 14 has been discontinued.

TABLE IV.—STEEL EXPOSURES AT KURE BEACH, N. C., 800-FT SITE, MARINE ATMOSPHERE.
Loss of Weight, g, 4 by 6-in. Steel Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]
1948.......	7.8	0.33	18.5	1.60	69.8	8.36	...	...
1949.......	11.5	1.54	25.5	4.44	54.4	8.52	(7 yr) 108.3	15.62
1950.......	10.6	0.60	21.6	1.20	44.1	5.95	...	...
1951.......	12.2	0.37	14.9	0.68	70.9	12.4	(5 yr) 100.7	11.36
1952.......	11.7	0.36	35.7	2.69	...	...	...	...
1953.......	17.5	2.11	51.7	9.56	...	...	(3 yr) 37.7	0.95
1954.......	12.7	0.25	24.2	0.98	...	...	...	...
1955.......	22.3	2.15	...	...	...	...	...	...
Average....	13.3	4.24[b]	27.4	11.60[b]	59.8	11.17[b]	...	...

[a] Standard Deviation of 4 specimens.
[b] Standard Deviation of the yearly losses.

TABLE V.—STEEL EXPOSURES AT KEARNY, N. J., INDUSTRIAL ATMOSPHERE.
Loss of Weight, g, 4 by 6-in. Steel Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]
1948........	34.5	0.96	41.4	0.59	56.1	0.46	(8 yr) 66.3	1.35
1949........	19.1	0.21	31.5	0.70	40.6	0.44	(7 yr) 45.7	1.87
1950........	39.5	2.47	47.9	1.37	51.2	3.33	(6 yr) 55.6	2.35
1951........	20.1	0.46	27.6	0.63	35.7	2.06	(5 yr) 38.7	7.85
1952........	16.5	0.13	22.5	0.31	28.2	0.50	...	...
1953........	14.8	0.19	19.5	0.26	...	...	(3 yr) 23.4	0.38
1954........	15.8	0.40	24.3	0.41	...	...	...	...
1955........	17.1	0.11	...	...	...	...	...	...
Average.....	22.2 20.4[c]	8.79[b] 4.82[c]	30.7 28.7[c]	9.67[b] 9.31[c]	42.4 ...	10.15[b] ...	... ...	... ...

[a] Standard Deviation of 4 specimens.
[b] Standard Deviation of the yearly losses.
[c] Omitting specimens exposed in 1948.

DISCUSSION—STEEL SPECIMENS

Examination of the data in Tables II to IX shows that the size of the average deviation of the samples of four specimens each was largely dependent upon the magnitude of the weight loss; that is, in most cases the greater the weight loss the greater the average deviation. The average deviations of the losses of the successive equal-exposure periods are higher than the deviation of the individual samples. This illustrates the effect of changes in climate during the various periods. The sites where these changes in corrosivity with time of exposure are most evident are New York, N. Y. (spring, Table III); Kure Beach, N. C., 800-ft site (Table IV); Kearny, N. J. (Table V); and New York, N. Y. (fall, Table VIII). Because of the wide difference in 1-yr losses of steels exposed at Kearny in 1948 and 1949 (Table V) and the fact that no specimens were exposed at New York (spring, Table III) in 1948, the averages for the exposures, omitting those made in 1948, are shown in parentheses in Table V. That the effects of weather are not extremely localized is shown by a comparison of the data in Tables III and V. Geographically, the

---

[3] Detailed information may be obtained from the National Research Council of Canada, Ottawa, Canada.

TABLE VI.—STEEL EXPOSURES AT MIDDLETOWN, OHIO,
SEMI-INDUSTRIAL ATMOSPHERE.
Loss of Weight, g, 4 by 6-in. Steel Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a
1948.......	9.1	0.12	14.4	0.26	22.1	0.21	(8 yr) 30.7	0.54
1949.......	9.2	0.09	16.1	0.24	21.3	0.15	(7 yr) 27.2	0.27
1950.......	10.9	0.15	13.6	0.32	19.6	0.29	(6 yr) 23.8	0.30
1951.......	8.2	0.05	12.1	0.15	17.5	0.14	(5 yr) 19.9	0.32
1952.......	7.5	0.11	10.8	0.25	16.8	0.14	...	...
1953.......	6.7	0.05	10.7	0.05	...	...	(3 yr) 14.0	0.16
1954.......	7.6	0.10	12.4	0.17	...	...	...	...
1955.......	8.1	0.17	...	...	...	...	...	...
Average....	8.4	1.22^b	12.9	1.82^b	19.5	2.06^b	...	...

[a] Standard Deviation of 4 specimens.
[b] Standard Deviation of the yearly losses.

TABLE VII.—STEEL EXPOSURES AT SOUTH BEND, PA.,
SEMI-RURAL ATMOSPHERE.
Loss of Weight, g, 4 by 6-in. Steel Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a
1948.......	10.8	0.12	16.9	0.13	28.6	0.23	(8 yr) 43.6	0.29
1949.......	10.0	0.05	19.3	0.17	28.2	0.61	(7 yr) 37.9	0.55
1950.......	13.8	0.12	18.9	0.26	29.8	0.27	(6 yr) 37.1	0.22
1951.......	9.8	0.09	15.5	0.09	23.9	0.23	(5 yr) 28.9	0.24
1952.......	9.9	0.09	17.2	0.26	24.8	0.15	...	...
1953.......	9.5	0.16	14.3	0.07	...	...	(3 yr) 19.8	0.11
1954.......	9.0	0.12	16.1	0.09	...	...	...	...
1955.......	11.1	0.21	...	...	...	...	...	...
Average....	10.5	1.40^b	16.9	1.79^b	27.1	2.29^b	...	...

[a] Standard Deviation of 4 specimens.
[b] Standard Deviation of the yearly losses.

test sites at Kearny and New York City are only a few miles apart. One is at ground level in the center of industrial activity in New Jersey; the other is on the roof on the 17-story New York Port Authority Building.

Attention is called to the very much larger losses of the specimens exposed at New York in the fall (Table VIII) and those exposed in the spring (Table III). Such effects of month of exposure have been previously noted.[4] These data show the necessity of exposing steel calibration specimens at approximately the same time of year at all locations. Attention is called to the difference in shapes of the time-corrosion curves between steels exposed in industrial atmosphere (Figs. 1, 2, and 3), and to the marine atmosphere (Fig. 7). The accelerated corrosion rate between 2- and 4-yr exposure is presumed to be caused by the accumulation of salts from evaporated sea-water spray on the groundward surfaces where it was not periodically removed by rain.

TABLE VIII.—STEEL EXPOSURES AT NEW YORK, N. Y., INDUSTRIAL ATMOSPHERE, FALL INSTALLATION.

Loss of Weight, g, 4 by 6-in. Steel Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]
1948	41.9	0.19	58.4	0.16	70.2	1.85	(8 yr) 78.9	1.98
1949	50.7	0.07	67.4	1.8	75.8	1.19	(7 yr) 84.4	4.07
1950	39.1	0.96	49.1	2.47	57.9	1.62	...	...
1951	44.0	1.51	53.9	1.51	60.9	0.72	(5 yr) 63.9	1.56
1952	43.7	0.80	51.8	0.22	60.7	2.52	...	...
1953	37.3	0.82	44.1	0.59	...	...	(3 yr) 47.6	0.36
1954	36.7	0.73	43.0	1.85	...	...	...	...
1955	30.5	0.69	...	...	...	...	...	...
Average	40.5	5.65[b]	52.5	7.69[b]	65.1	6.77[b]	...	...

[a] Standard Deviation of 4 specimens.
[b] Standard Deviation of the yearly losses.

In Table XX is shown the relative corrosivity of the atmosphere at 19 test sites. The losses of weight at State College, Pa., for each of the four periods employed (1, 2, 4, and 8 yr) are used as unity. In many cases the relative corrosivity is approximately the same whether based upon 1-, 2-, or 4-yr exposures. The relative corrosivity of the marine atmospheres tends to increase with length of exposure, whereas that in an industrial atmosphere, particularly the fall exposure at New York City, tends to decrease with length of exposure. This is also shown graphically in Figs. 1 to 6 for several sites where the data were complete.

The data in Table XX show that steel corroded over 600 times as much in 2 yr at the 80-ft site at Kure Beach, N. C. (severe marine) as at Norman Wells, N. W. T. (polar), or 40 times that at Esquimalt, Vancouver Island, B. C., during a similar period. The test site at Esquimalt is located 1500 ft from the water at an elevation of 50 ft; however, the prevailing wind tends to prevent the deposition of sea-water spray on the specimens. Therefore, the atmosphere can be considered rural (corrosivity one half that at State College).

---

[4] G. N. Schramm and E. S. Taylerson, "The Influence of Rainfall and Smoke on the Corrosion of Iron and Steel," Symposium on Outdoor Test Results on Bare and Metal-Coated Ferrous Specimens, Am. Soc. Testing Mats., p. 51 (1934). (Issued as separate publication *ASTM STP No. 17.*)

TABLE IX.—STEEL EXPOSURES AT FOURTEEN SITES.
Loss of Weight, g, 4 by 6-in. Steel Specimens.

Year Exposed	Norman Wells, N.W.T.				Trail, B.C.		Vancouver, B.C.				Saskatoon, Sask.				Montreal, Que.				Ottawa, Ont.			
	1 yr		2 yr		1 yr		1 yr		2 yr		1 yr		2 yr		1 yr		2 yr		1 yr		2 yr	
	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a
1954	0.30	0.05	0.30	0.00	...	...	3.3	0.12	5.8	0.22	4.7	0.15	7.3	0.09	11.5	0.95	15.7	0.59	7.9	0.23	11.2	0.17
1955	0.05	0.01	...	...	8.8	0.13	3.1	0.09	...	...	4.3	0.07	...	...	8.6	0.01	...	...	6.6	0.07	...	...
1956	0.32	0.05	...	...	10.8	0.12	3.4	0.07	...	...	3.3	0.10	...	...	9.8	0.12	...	...	6.9	0.02	...	...
Average	0.22	0.12^b	0.30		9.8	0.10^b	3.3	0.14^b	5.8		4.1	0.59^b	7.3		10.0	1.19^b	15.7		7.1	0.55^b	11.2	

Year Exposed	Halifax, Federal Building, N.S.				Halifax, York Redoubt, N.S.				Perrine, Fla.						Daytona Beach, Fla.					
	1 yr		2 yr		1 yr		2 yr		1 yr		2 yr		4 yr		1 yr		2 yr		4 yr	
	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a
1951	...	...	...	...	...	...	...	...	5.9	0.09	9.4	0.07	12.1	0.21	48.4	1.52	133.7	2.12	331.6	10.75
1952	...	...	...	...	...	...	...	...	6.6	0.16	7.9	0.07	...	...	34.9	7.25	134.0	6.53	345.7	0.85
1953	...	...	...	...	...	...	...	...	...	...	7.7	0.18	...	...	68.6	4.35	180.5	7.03	...	...
1954	28.0	0.54	42.4	0.57	11.3	0.44	17.4	0.93	4.9	0.16	...	...	...	...	34.9	13.24	141.3	9.62	...	...
1955	24.0	0.39	...	...	9.7	0.21	...	...	5.8	0.07	...	...	...	...	55.6	13.74	...	...	...	...
1956	25.5	0.27	...	...	9.8	0.09	...	...	...	...	...	...	...	...	...	...	...	...	...	...
Average	25.8	1.70^b	42.4		10.3	0.73^b	17.4		5.8	0.60^b	8.3	0.76^b	12.1		48.5	14.36^b	147.4	19.36^b	338.7	7.05^b

Year Exposed	Sandy Hook, N. J.				Point Reyes, Calif.						Kure Beach, N. C. (80 ft site)						Kure Beach, N. C. (800 ft site) 0.187-in. thick specimens					
	1 yr		2 yr		1 yr		2 yr		4 yr		1 yr		2 yr		4 yr		1 yr		2 yr		4 yr	
	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a
1948	15.6	0.14	23.1	0.33	...	...	...	...	...	...	...	...	...	...	84.3	5.09	...	...	...	...	...	...
1949	13.2	0.09	23.5	0.21	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
1950	16.8	0.16	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
1951	...	...	...	...	...	...	...	...	...	...	23.4	1.05	121.0	13.30	417.9	^{c}	12.0	0.49	12.4	0.36	36.3	2.07
1952	...	...	...	...	12.0	0.19	23.1	0.78	39.2	1.95	94.1	10.24	237.2	17.69	356.2	0.44^d	11.0	0.41	20.7	1.19	42.4	5.35
1953	...	...	...	...	12.9	0.12	23.0	0.65	...	...	121.7	8.50	378.6	59.69	...	...	11.4	0.80	27.0	1.87	...	...
1954	...	...	...	...	12.7	0.38	16.5	0.56	...	...	108.4	5.82	236.7	15.06	...	...	10.8	0.39	17.6	0.93	...	...
1955	...	...	...	...	11.0	0.32	...	...	...	...	103.2	8.68	...	...	...	...	14.8	1.01	...	...	...	...
1956	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
Average	15.2	1.50^b	23.3	0.14^b	12.2	0.74^b	20.9	3.55^b	39.2		90.2	34.55^b	218.4	94.65^b	387.1	30.85^b	12.0	1.46^b	19.4	5.41^b	39.4	3.05^b

[a] Standard Deviation of 4 specimens.
[b] Standard Deviation of the yearly losses.
[c] One Sample.
[d] Two Samples.

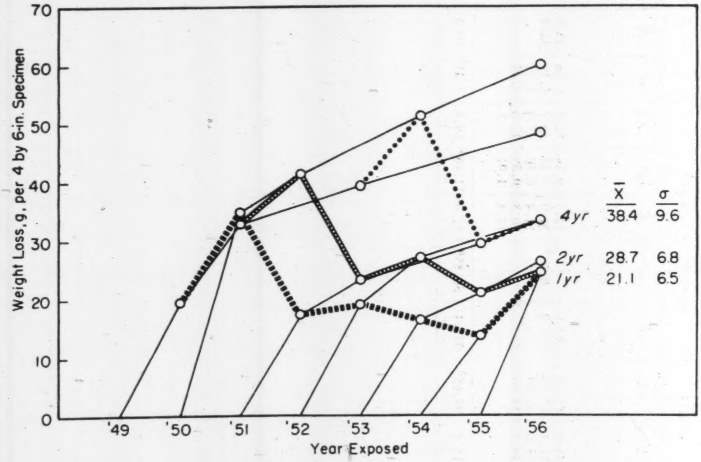


FIG. 1.—Graphical Presentation of Complete Data on Steel, New York, N. Y. (Spring Exposure).

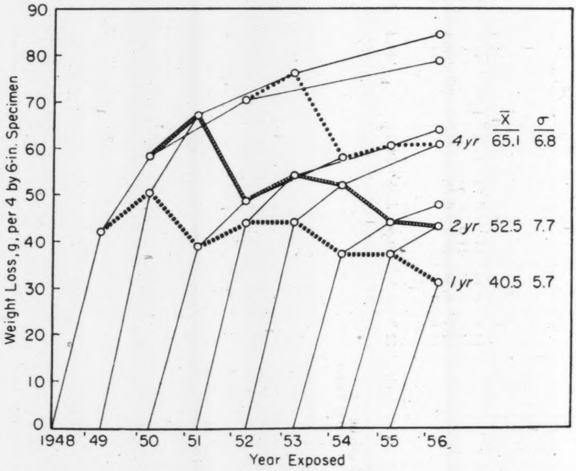


FIG. 2.—Graphical Presentation of Complete Data on Steel, New York, N. Y. (Fall Exposure).

The large difference in corrosivity of the two test sites in Halifax, N. S. (sites 10 and 17, Table XX) is caused by the fumes from a smoke stack which is near the test rack at site 17.

Conclusions:

1. The 4 by 6-in. steel specimens are a satisfactory material to calibrate the corrosivity of various atmospheric test sites toward steel.

2. At rural and mild industrial sites a single 1-yr test will be sufficient to indicate the relative corrosivity.

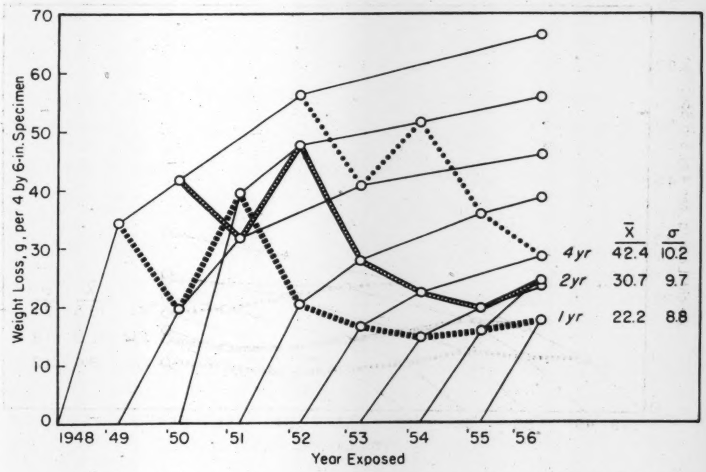


FIG. 3.—Graphical Presentation of Complete Data on Steel, Kearny, N. J.

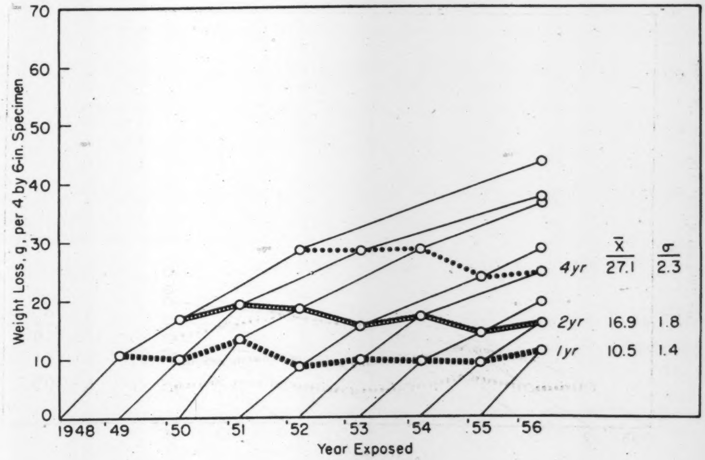


FIG. 4.—Graphical Presentation of Complete Data on Steel, South Bend, Pa.

3. At a severe industrial site, at least three successive yearly exposures may be necessary to obtain a reliable index.

4. At marine sites exposures longer than 1 yr are required to obtain a realistic figure. Three years seems to be sufficient at sites where deposition of seawater spray on the specimens is frequent but there is a large difference in "meas-

ured" corrosivity between 4- and 8-yr exposures where the spray conditions are not so severe.

X to XIX. An examination of these data shows that there was considerable variation in the weight losses of zinc samples exposed at the same location at different times. These variations point out the effect of changes in weather on corrosion rates.

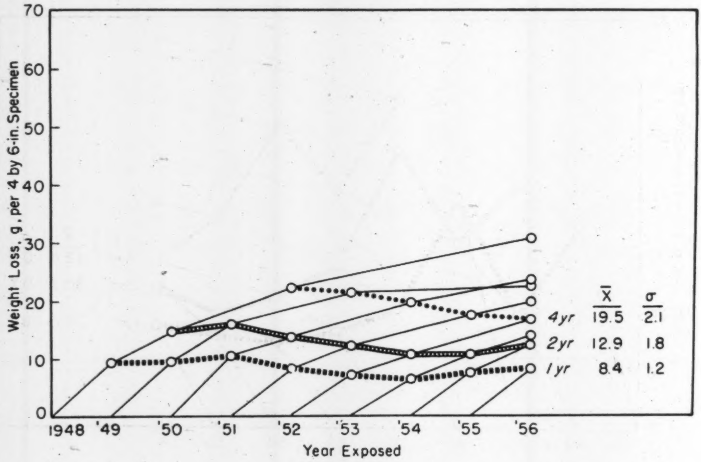


FIG. 5.—Graphical Presentation of Complete Data on Steel, Middletown, Ohio.

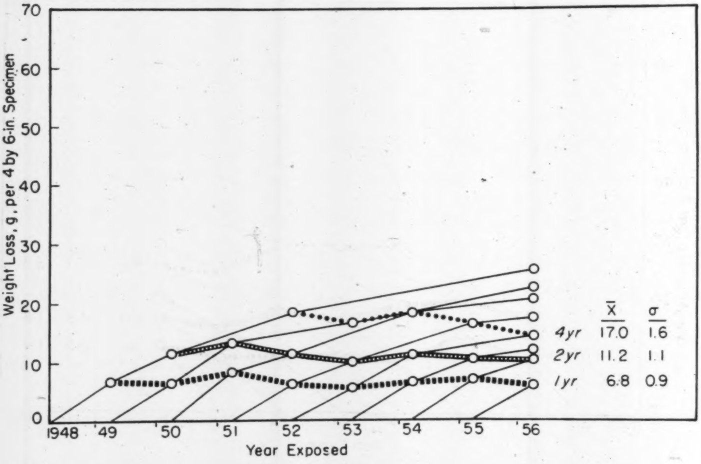


FIG. 6.—Graphical Presentation of Complete Data on Steel, State College, Pa.

DISCUSSION—ZINC SPECIMENS

The complete weight loss data for all of the zinc samples are given in Tables

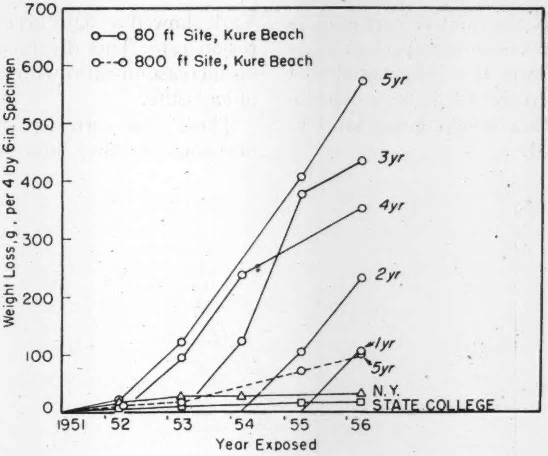


FIG. 7.—Time *Versus* Corrosion Curves for Steel at Kure Beach (80- and 800-ft sites), N. C., New York, N. Y. (Spring), and State College, Pa.

TABLE X.—ZINC EXPOSURES AT STATE COLLEGE, PA., RURAL ATMOSPHERE.
Loss of Weight, g, 4 by 6-in. Zinc Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a
1948..........	0.364	0.050	0.476	0.026	1.198	0.037	(8 yr) 1.874	0.044
1949..........	0.281	0.005	0.549	0.008	0.994	0.006	(7 yr) 1.675	0.048
1950..........	0.244	0.009	0.442	0.010	0.804	0.008	(6 yr) 1.210	0.011
1951..........	0.269	0.012	0.476	0.014	0.900	0.039	...	...
1952..........	0.258	0.025	0.464	0.036	0.887	0.021	...	...
1953..........	0.366	0.012	0.595	0.006	...	...	(3 yr) 0.878	0.004
1954..........	0.366	0.012	0.631	0.017	...	...	...	...
1955..........	0.334	0.008	...	...	...	...	...	...
Average.......	0.3102	0.0166	0.5190	0.0167	0.9566	0.0222	...	...

[a] Standard Deviation of 4 specimens.

TABLE XI.—ZINC EXPOSURES AT NEW YORK, N. Y., INDUSTRIAL ATMOSPHERE, SPRING INSTALLATION.
Loss of Weight, g, 4 by 6-in. Zinc Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a
1949.........	1.199	0.041	2.504	0.061	5.847	0.172	(8 yr) 12.041	0.285
1950.........	1.072	0.006	2.420	0.033	5.349	0.010	(7 yr) 11.529	0.354
1951.........	1.054	0.002	2.399	0.021	5.256	0.075	(6 yr) 9.109	0.060
1952.........	1.166	0.032	2.289	0.036	5.606	0.165	...	...
1953.........	0.888	0.012	2.029	0.080	...	...	...	...
1954.........	1.068	0.020	2.743	0.081	...	...	(3 yr) 3.769	0.033
1955.........	1.392	0.002	...	...	...	...	...	...
Average......	1.1198	0.0164	2.3973	0.0523	5.5145	0.1055	...	...

[a] Standard Deviation of 4 specimens.

In Table XX the relative corrosivities of the atmospheres with respect to State College are shown. It will be noted that the relative corrosivities do vary depending upon whether weight losses for 1 yr or 2 yr are used.

Some of the ratios differ little, such as South Bend, Pa. Others differ by considerable amounts, such as Kure Beach or New York. One reason for these differences becomes evident upon inspection of time-corrosion curves (Fig. 8).

State College, upon which these ratios are based, showed a slight decrease in corrosion rate after the first year. New York showed a slight acceleration in corrosion rate. This divergence resulted in an increase in ratio with increasing time of exposure.

These time-corrosion curves (Fig. 8) also show another interesting fact. New York, Kearny, Sandy Hook, South Bend, and Middletown—all predominantly with industrial contamination—show almost linear corrosion rates. Kure Beach, Daytona, Point Reyes, and Perrine—all with some degree of marine contamination—show decreases in corrosion rates after the first year. Thus, these latter atmospheres tend to show less cor-

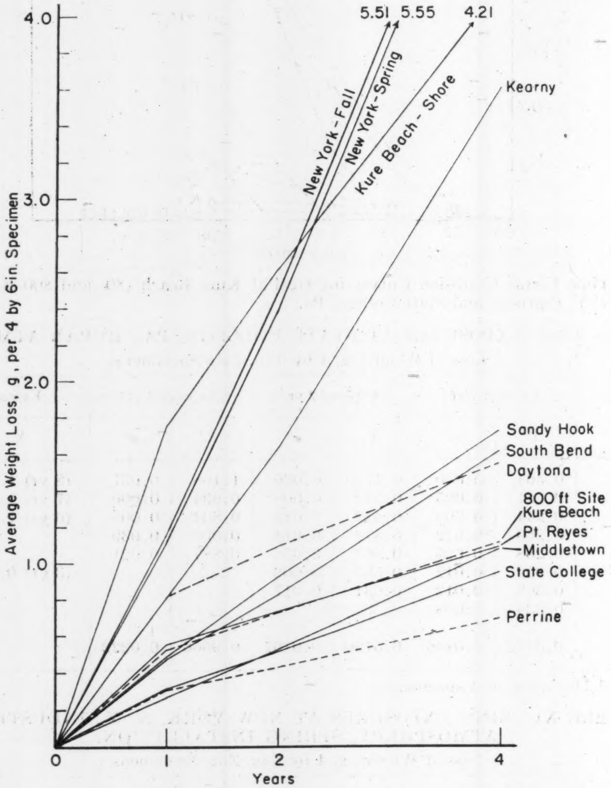


FIG. 8.—Time *Versus* Corrosion Curves for Zinc at the United States Test Sites.

TABLE XII.—ZINC EXPOSURES AT KURE BEACH, N. C., MARINE ATMOSPHERE.

Loss of Weight, g, 4 by 6-in. Zinc Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 3 yr		Exposed 4 yr		Exposed to 1956	
	$\overline{X}$	σ^a	$\overline{X}$	σ^a	$\overline{X}$	σ^a	$\overline{X}$	σ^a	$\overline{X}$	σ^a
					800 FT SITE					
1948	0.533	0.045	0.644	0.044	...	...	1.082	0.046	(8 yr) 2.035	0.067
1949	0.501	0.013	0.707	0.033	...	...	1.147	0.043	(7 yr) 1.871	0.062
1950	0.434	0.020	0.687	0.039	...	...	1.184	0.061	(6 yr) 1.724	0.066
1951	0.433	0.007	0.738	0.008	...	...	1.248	0.056	...	...
1952	0.370	0.014	0.692	0.038	...	...	1.184	0.069	...	...
1953	0.628	0.034	0.830	0.058	1.205	0.073	...	...	...	...
1954	0.551	0.020	0.802	0.026	...	...	...	...	...	...
1955	0.753	0.031	...	...	...	...	...	...	...	...
Average	0.5254	0.0230	0.7286	0.0351	1.205	0.073	1.1690	0.0550	...	...
					80 FT SITE, BEACH EXPOSURE					
1948	1.882	0.141	2.114	0.016	...	...	3.332	0.037	(8 yr) 7.713	[d]
1949	1.156	0.016	2.909	0.041	...	...	3.273	0.138	(7 yr) 6.552	0.270[c]
1950	1.361	0.009	2.086	0.040	2.714[b]	0.037	...	...	(6 yr) 6.333	0.334
1951	1.160	0.028	1.928	0.020	...	...	5.230	0.312	...	...
1952	2.976	0.262	2.831	0.126	...	...	4.995	0.606[c]	...	...
1953	2.134	0.040	3.794	0.150	4.278	0.120[c]	...	...	...	...
1954	1.961	0.090	2.659	0.026	...	...	...	...	...	...
1955	1.446	0.079	...	...	...	...	...	...	...	...
Average	1.7595	0.0831	2.6173	0.0598	3.4960	0.0785	4.2075	0.2732	...	...

[a] Standard Deviation of 4 specimens.
[b] Removed in 1953 through error.
[c] Range of 2 specimens, 2 lost in hurricane Hazel.
[d] One specimen only.

TABLE XIII.—ZINC EXPOSURES AT KEARNY, N. J., INDUSTRIAL ATMOSPHERE.

Loss of Weight, g, 4 by 6-in. Zinc Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\overline{X}$	σ^a	$\overline{X}$	σ^a	$\overline{X}$	σ^a	$\overline{X}$	σ^a
1948	1.106	0.054	1.738	0.029	3.834	0.043	(8 yr) 7.921	0.346
1949	0.768	0.010	1.637	0.030	3.688	0.041	(7 yr) 6.863	0.097
1950	0.788	0.014	1.720	0.025	3.691	0.087	(6 yr) 5.901	0.130
1951	0.794	0.012	1.637	0.025	3.612	0.088	...	...
1952	0.818	0.012	1.598	0.012	3.334	0.174	...	...
1953	0.740	0.006	1.559	0.018	...	...	(3 yr) 2.481	0.012
1954	0.743	0.010	1.533	0.012	...	...	...	...
1955	0.727	0.009	...	...	...	...	...	...
Average	0.8105	0.0159	1.6317	0.0216	3.6318	0.0866	...	...

[a] Standard Deviation of 4 specimens.

TABLE XIV.—ZINC EXPOSURES AT MIDDLETOWN, OHIO, SEMI-INDUSTRIAL ATMOSPHERE.

Loss of Weight, g, 4 by 6-in. Zinc Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a
1948..........	0.320	0.028	0.539	0.007	1.152	0.001	(8 yr) 2.334	0.022
1949..........	0.302	0.013	0.602	0.022	1.195	0.041	(7 yr) 2.086	0.047
1950..........	0.278	0.017	0.567	0.013	1.077	0.015	(6 yr) 1.680	0.016
1951..........	0.268	0.006	0.514	0.012	1.021	0.019	...	...
1952..........	0.249	0.006	0.488	0.007	0.987	0.011	...	...
1953..........	0.233	0.011	0.488	0.019	...	...	(3 yr) 0.787	0.010
1954..........	0.230	0.004	0.489	0.006	...	...	...	...
1955..........	0.264	0.018	...	...	...	...	...	...
Average.......	0.2680	0.0129	0.5267	0.0123	1.0864	0.0174	...	...

[a] Standard Deviation of 4 specimens.

TABLE XV.—ZINC EXPOSURES AT SOUTH BEND, PA., SEMI-RURAL ATMOSPHERE.

Loss of Weight, g, 4 by 6-in. Zinc Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a
1948..........	0.599	0.074	0.792	0.039	1.700	0.009	(8 yr) 3.387	0.077
1949..........	0.332	0.008	0.731	0.006	1.561	0.022	(7 yr) 2.715	0.036
1950..........	0.534	0.017	0.968	0.016	1.646	0.016	(6 yr) 2.536	0.054
1951..........	0.372	0.010	0.740	0.035	1.384	0.093	...	...
1952..........	0.566	0.023	0.885	0.009	1.811	0.036	...	...
1953..........	0.506	0.018	0.909	0.020	...	...	(3 yr) 1.382	0.019
1954..........	0.352	0.017	0.748	0.023	...	...	...	...
1955..........	0.528	0.012	...	...	...	...	...	...
Average.......	0.4735	0.0224	0.8247	0.0211	1.6204	0.0352	...	...

[a] Standard Deviation of 4 specimens.

TABLE XVI.—ZINC EXPOSURES AT NEW YORK, N. Y., INDUSTRIAL ATMOSPHERE, FALL INSTALLATION.

Loss of Weight, g, 4 by 6-in. Zinc Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 4 yr		Exposed to 1956	
	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a	$\bar{X}$	σ^a
1948..........	1.157	0.089	2.642	0.035	5.769	0.133	...	...
1949..........	1.442	0.018	2.769	0.041	5.808	0.083	(7 yr) 10.655	0.081
1950..........	1.203	0.015	2.536	0.039	5.546	0.147	(6 yr) 8.919	0.223
1951..........	1.043	0.017	2.337	0.019	5.343	0.059	(5 yr) 6.682	0.072
1952..........	1.128	0.017	2.191	0.022	5.272	0.042	...	...
1953..........	0.964	0.016	2.608	0.012	...	...	(3 yr) 3.790	0.083
1954..........	1.284	0.018	2.330	0.024	...	...	...	...
1955..........	1.054	0.010	...	...	...	...	...	...
Average.......	1.1594	0.0250	2.4876	0.0274	5.5476	0.0928	...	...

[a] Standard Deviation of 4 specimens.

TABLE XVII.—ZINC EXPOSURES IN MARINE ATMOSPHERES IN FLA. AND CALIF.

Loss of Weight, g, 4 by 6-in. Zinc Specimens.

Placed on Exposure	1 yr		2 yr		4 yr		Exposed to 1956	
	$\overline{X}$	σ[a]	$\overline{X}$	σ[a]	$\overline{X}$	σ[a]	X	σ[a]
DAYTONA BEACH, FLA.								
1949	1.247	0.070	1.542	0.120	1.719	0.026	(7 yr) 2.743	0.034
1950	0.746	0.026	1.080	0.008	1.408	0.055	(6 yr) 2.027	0.010
1951	0.630	0.027	0.854	0.024	1.496	0.045	...	...
1952	0.641	0.005	0.927	0.019	1.584	0.061	...	...
1953	0.719	0.047	0.979	0.032	...	...	(3 yr) 1.315	0.031
1954	0.826	0.007	1.199	0.022	...	...	...	...
1955	0.910	0.035	...	...	...	...	...	...
Average	0.8170	0.0310	1.0968	0.0375	1.5518	0.0468	...	...
PERRINE, FLA.								
1951	0.340	0.039	0.392	0.028	0.724	0.038	(5 yr) 0.918	0.050
1952	0.228	0.020	0.372	0.026	0.675	0.008	...	...
1953	...	...	0.470	0.023	...	...	(3 yr) 0.613	0.017
1954	0.347	0.022	0.488	0.007	...	...	...	...
1955	0.311	0.006	...	...	...	...	...	...
Average	0.3065	0.0218	0.4305	0.0210	0.700	0.023	...	...
POINT REYES, CALIF.								
1952	0.570	0.022	0.716	0.051	1.095	0.023	...	...
1953	0.588	0.015	0.776	0.019	...	...	(3 yr) 0.924	0.014
1954	0.562	0.034	0.717	0.068	...	...	...	...
1955	0.493	0.025	...	...	...	...	...	...
Average	0.5532	0.0240	0.7363	0.0460	1.095	0.023	...	...

[a] Standard Deviation of 4 specimens.

TABLE XVIII.—ZINC EXPOSURES AT SANDY HOOK, N. J., INDUSTRIAL ATMOSPHERE.*

Loss of Weight, g, 4 by 6-in. Zinc Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 3 yr		Exposed 4 yr[a]	
	$\overline{X}$	σ[a]	$\overline{X}$	σ[a]	$\overline{X}$	σ[a]	$\overline{X}$	σ[a]
1948	0.494	0.025	0.797	0.014	...	...	1.721 1.748	0.006[b] 0.009
1949	0.521	0.008	0.970	0.022	1.533 1.531	0.038 0.028		
1950	0.463	0.006	1.013 1.002 0.996 1.004	0.021 0.023 0.022 0.022	...	...	...	...
1951	0.468 0.460 0.442 0.457	0.009 0.003 0.043 0.018	...	...	...	...	...	...
Average	0.4838	0.014	0.9237	0.019	1.532	0.033	1.734	0.008

* All specimens removed in 1952.

[a] Standard Deviation of 4 specimens.

[b] Range for 2 specimens.

rosivity with time, whereas the others show little change.

However, these data do show, for the most part, some consistency from year to year. The bar-graph (Fig. 9) indicates the ranges of weight losses observed throughout the test. Except for the 80-ft site Kure Beach exposure, the total variation from year to year is not great. These variations are enough to make it doubtful that data from a single 1-yr exposure would be an acceptable criterion for atmospheric calibration.

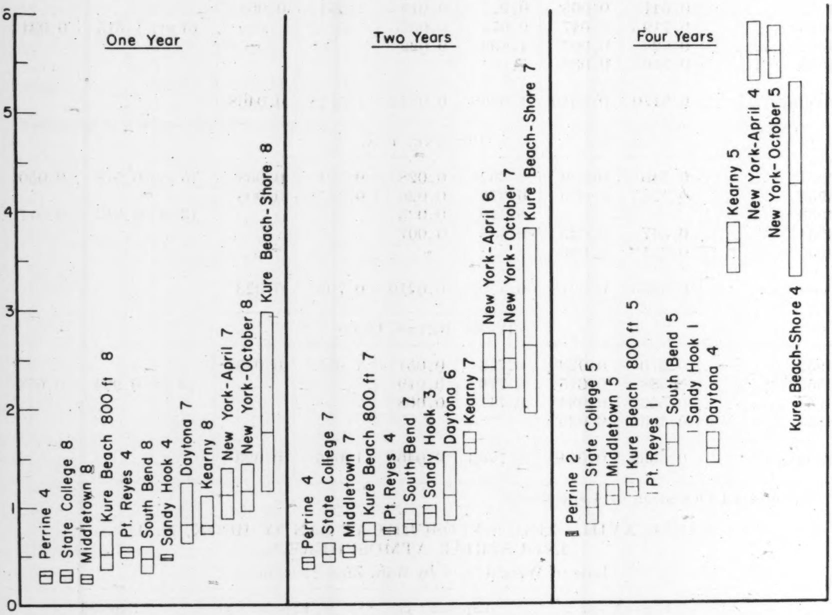


FIG. 9.—Range of Weight Losses Observed Throughout the Test for Zinc at the United States Test Sites.

In order to reduce this variation, data from three successive years were averaged. That is, samples exposed in 1948, 1949, and 1950 were averaged; 1949, 1950, and 1951; etc. This resulted in some improvement of the variations in 1 yr weight losses.

The 2 yr weight losses were similarly averaged. Here the improvement is greater than with the 1 yr data.

Conclusions:

1. Zinc specimens, 4 by 6 in. in size, are satisfactory as a material with which to calibrate the corrosivity of various atmospheric test sites *with respect to zinc.*

2. At least three successive 2-yr exposures may be necessary to obtain a relative corrosion index.

3. At marine locations, because of the decrease in corrosion rates with time, it is even more important to use 2-yr weight-loss data than 1-yr weight-loss data.

COMPARISONS BASED ON BOTH STEEL AND ZINC

Examination of the data in Table XX shows that in many cases the relative corrosivity of atmospheres based on losses of weight of steel is in reasonably good agreement with that based on losses of weight of zinc. There are notable exceptions, the most outstanding of which are sites 17 and 19 (Halifax Federal Building and 80-ft site at Kure Beach). At site 17 in two years, zinc was corroded 22 times that at State College, whereas steel was corroded less than 4 times as much. Conversely, at site 19 zinc was corroded 5 times that at State College, whereas steel was corroded 20 times as much. Other reversals were found. This emphasizes that the corrosivity of the atmosphere toward one metal cannot be estimated with certainty by observations on another metal.

TABLE XIX.—ZINC EXPOSURES AT CANADIAN RESEARCH COUNCIL TEST SITES

Loss of Weight, g, 4 by 6-in. Zinc Specimens.

Placed on Exposure	Exposed 1 yr		Exposed 2 yr		Exposed 1 yr		Exposed 2 yr	
	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]	$\bar{X}$	σ[a]
	OTTAWA, ONT.				SASKATOON, SASK.			
1954	0.367	0.012	0.672	0.010	0.207	0.011	0.311	0.012
1955	0.460	0.099	...	...	0.146	0.006	...	...
1956	0.280	0.005	...	...	0.112	0.003	...	...
Average	0.3690	0.009	...	...	0.1550	0.007	...	...
	MONTREAL, QUE.				HALIFAX, FEDERAL BUILDING, N. S.			
1954	0.729	0.011	1.546	0.034	5.831	0.189	11.555	0.189
1955	0.714	0.012	...	...	6.734	0.008	...	...
1956	0.647	0.015	...	...	4.186	0.145	...	...
Average	0.6967	0.013	...	...	5.5837	0.1140	...	...
	HALIFAX, YORK REDOUBT, N. S.				NORMAN WELLS, N. W. T.			
1954	0.609	0.007	1.081	0.003	0.154	0.018	0.167	0.006
1955	0.474	0.012	...	...	0.084	0.006	...	...
1956	0.435	0.020	...	...	0.085	0.013	...	...
Average	0.5060	0.013	...	...	0.1077	0.012	...	...
	ESQUIMALT, B. C.				TRAIL, B. C.			
1954	0.107	0.002	0.201	0.003	...	...	...	...
1955	0.142	0.004	...	...	0.454	0.017	...	...
1956	0.155	0.002	...	...	0.519	0.004	...	...
Average	0.1343	0.003	...	...	0.4865	0.010	...	...

[a] Standard Deviation of 4 specimens.

GENERAL CONCLUSIONS

Consideration of the data from both steel and zinc specimens shows that in some atmospheres there are decided differences in the relative performances of steel and of zinc and, consequently, of the relative corrosivity as measured by losses of weight of those metals. It follows that the relative corrosivity of atmospheres might be different if still

TABLE XX.—RELATIVE CORROSIVITY OF ATMOSPHERE AT NINETEEN TEST SITES WITH THOSE AT STATE COLLEGE, PA., AS UNITY.

Based on Losses of 4 by 6-in. Specimens.

Rural Exposure Sites—Nos. 1, 2, 3, 4, 5, 10, 11.
Industrial Exposure Sites—Nos. 6, 7, 8, 9, 15, 16, 17.
Marine Exposure Sites—Nos. 12, 13, 14, 18, 19.

Site	Location	Steel Exposed				Zinc Exposed			
		1 yr	2 yr	4 yr	8 yr	1 yr	2 yr	4 yr	8 yr
1....	Norman Wells, N.W.T..........	0.03 (3)[a]	0.03 (1)[a]	...	...	0.4 (3)[a]	0.3 (1)[a]	...	...
2....	Esquimalt, Vancouver Island, B.C..............	0.5 (3)	0.5 (1)	...	...	0.4 (3)	0.4 (1)	...	...
3....	Saskatoon, Sask....	0.6 (3)	0.7 (1)	...	...	0.5 (3)	0.6 (1)	...	...
4....	Perrine, Fla.[b]......	0.9 (4)	0.7 (3)	0.7 (1)[a]	...	1.0 (4)	0.8 (4)	0.7 (2)[a]	...
5....	State College, Pa...	*1.0* (8)	*1.0* (7)	*1.0* (1)	*1.0* (1)[a]	*1.0* (8)	*1.0* (7)	*1.0* (5)	*1.0* (1)[a]
6....	Ottawa, Ont........	1.0 (3)	1.0 (1)			1.2 (3)	1.3 (1)		
7....	Middletown, Ohio..	1.2 (8)	1.2 (7)	1.2 (5)	1.2 (1)	0.9 (8)	1.0 (7)	1.1 (5)	1.2 (1)
8....	Trail, B. C.........	1.4 (2)				1.6 (2)			
9....	Montreal, Que.......	1.5 (3)	1.4 (1)	...	...	2.2 (3)	3.0 (1)	...	...
10....	Halifax (York Redoubt), N.S......	1.5 (3)	1.6 (1)	...	...	1.6 (3)	2.1 (1)	...	...
11....	South Bend, Pa.[c]...	1.5 (8)	1.5 (7)	1.6 (5)	1.7 (1)	1.5 (8)	1.6 (7)	1.7 (5)	1.8 (1)
12....	800-ft site, Kure Beach, N. C. 0.187-in. Steel[d].........	1.8 (5)	1.7 (4)	2.3 (2)	2.4 (5 yr)	1.7 (8)	1.4 (7)	1.2 (5)	1.1 (1)
13....	Point Reyes, Calif..	1.8 (4)	1.9 (3)	2.3 (1)	...	1.8 (4)	1.4 (4)	1.1 (1)	...
12....	800-ft site, Kure Beach, N. C. 0.065-in. Steel..........	2.0 (8)	2.5 (7)	3.5 (4)	5.8 (5 yr)	...	...	...	...
14....	Sandy Hook, N. J..	2.2 (3)	2.1 (2)	...	...	1.6 (4)	1.8 (3)	1.8 (1)	...
15....	New York, N. Y. (Spring)..........	3.1 (7)	2.6 (6)	2.3 (4)	2.1 (7 yr)	3.6 (7)	4.6 (6)	5.8 (4)	6.4 (1)
16....	Kearny, N. J.......	3.3 (8)	2.7 (7)	2.5 (5)	2.6 (1)	2.6 (8)	3.1 (7)	3.8 (5)	4.2 (1)
17....	Halifax (Federal Building), N.S....	3.8 (3)	3.8 (1)	...	...	18 (3)	22 (1)	...	...
15....	New York, N. Y. (Fall)...........	6.0 (8)	4.7 (7)	3.8 (5)	3.1 (1)	3.7 (8)	4.8 (7)	5.8 (5)	5.7 (1)
18....	Daytona Beach, Fla.	7.1 (5)	13 (4)	20 (2)	...	2.6 (7)	2.1 (6)	1.6 (4)	1.6 (7 yr)
19....	80-ft site, Kure Beach, N. C. 0.187-in. Steel[d]...	13 (5)	20 (4)	23 (2)	...	5.7 (8)	5.0 (7)	4.4 (4)	4.1 (1)

[a] Numeral in ( ) indicates number of such periods.
[b] 20 miles southwest of Miami, Fla. 5 miles from ocean.
[c] 36 miles northeast of Pittsburgh, Pa.
[d] Used after 0.062-in. specimens showed excessive corrosion.

other metals were used as calibration specimens; therefore, it is necessary to state the metal used when reporting relative corrosivity of atmospheres.

FUTURE WORK

The Advisory Committee on Corrosion has requested Committee B-3 to determine the relative corrosivity of certain

ASTM test sites that were not included in the present work.

It is planned to expose a new series specimens in the spring of 1960 at these sites, as well as about 25 others. State College will again be used as unity for purposes of comparing the two sets of data. There will be four 1-yr exposures, three 2-yr exposures, and one 4-yr exposure. Because of the demonstrated good reproducibility of replicate specimens, two specimens will constitute a "sample."

REPORT OF COMMITTEE B-4
ON
METALLIC MATERIALS FOR ELECTRICAL HEATING, ELECTRICAL RESISTANCE, AND ELECTRICAL CONTACTS*

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts held three meetings during the year: in Boston, Mass., on June 26, 1958; in Chicago, Ill., on October 24, 1958; and in Washington, D. C., on February 25, 1959.

The committee consists of 57 members, of whom 23 are classified as producers, 24 as consumers, and 10 as general interest members.

As it has done for the past several years, Committee B-4 cooperated again this year with Pennsylvania State University in an Engineering Seminar on Electrical Contacts. This symposium was held from June 8 to June 12, 1959.

The committee continues to sponsor the Bibliography and Abstracts on Electrical Contacts published as STP 56 G and annual supplements. In 1958, the society published supplement STP 56 L covering the literature abstracts for 1957. This bibliography now covers all pertinent literature references on electrical contacts from 1835 to and including 1957.

At the meeting held in Chicago on October 24, 1958, Mr. Leonard Mayeron was elected Vice-Chairman of Committee B-4.

At the meeting held in Washington on February 25, 1959, Dr. Ragnar Holm was elected to honorary membership on Committee B-4 for outstanding service to the activities of Subcommittee IV on Contact Materials. The other honorary members of this committee are: Messrs. P. H. Brace, F. E. Carter, and R. D. Van Nordstrand.

The committee was grieved to learn of the sudden death of one of its members, Mr. D. K. Wells. Mr. Wells had been very active in the work of Subcommittee IV and had served at one time as chairman of Section C of Subcommittee IV.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee B-4 presented to the Society through the Administrative Committee on Standards the Tentative Method of Test for Surety of Make of Electrical Contact Materials (B 340 – 59 T). This recommendation was accepted by the Standards Committee on March 25, 1959.

NEW STANDARD,
IMMEDIATE ADOPTION

The committee recommends that the Specifications for Drawn or Rolled Nickel-Chromium and Nickel-Chromium-Iron Alloys for Electrical Heating Elements, as appended hereto,[1] be approved for reference to letter ballot of the Society for adoption as standard,

---

*** Sixty-second Annual Meeting of the Society, June 21–26, 1959.**

[1] The new standard appears in the 1959 Supplement to Book of ASTM Standards, Part 2.

and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the specifications may be referred to letter ballot of the Society.

New Tentative

The committee recommends for publication as tentative the Method of Accelerated Life Test of Iron-Chromium-Aluminum Alloys for Electrical Heating, as appended hereto.[2]

Revision of Standard, Immediate Adoption

The committee recommends for immediate adoption revisions as follows of the Standard Method of Accelerated Life Test for Metallic Materials for Electrical Heating (**B 76 – 57**),[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Title.—Revise to read "Standard Method of Accelerated Life Test of Nickel-Chromium and Nickel-Chromium-Iron Alloys for Electrical Heating."

Section 1.—Revise to read as follows:

> 1. This method covers the determination of the resistance to oxidation of nickel-chromium and nickel-chromium-iron electrical heating alloys at elevated temperatures under intermittent heating.

Section 3 (*g*).—Delete all of Paragraph (*g*) except the first sentence.

Section 7 (*c*).—Following the temperature of test given for 80 per cent nickel and 20 per cent chromium alloy, add the following: "for alloys of nominal composition 35 per cent nickel, 20 per cent chromium, and 45 per cent iron, the temperature of test shall be 1950 F (1066 C)."

Withdrawal of Standards

The committee recommends the withdrawal of the following standards:

Standard Specifications for:

Drawn or Rolled Alloy, 80 per cent Nickel, 20 per cent Chromium, for Electrical-Heating Elements (**B 82 – 57**), and

Drawn or Rolled Alloy, 60 per cent Nickel, 16 per cent Chromium, and Balance Iron, for Electrical-Heating Elements (**B 83 – 52**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

Activities of Subcommittees

As in the past, the major part of the activities of the subcommittees involves research into the operating properties of materials under the purview of the committee as well as the engineering problems concerned with providing reproducible testing procedures. The results of this research activity become evident only in the tentatives and standards which come from the group. However, the benefits to the individual members coming from the discussions go beyond the immediate problems of this activity. We feel that this is one of the main reasons why the meetings of the subcommittees, as well as the sections of the subcommittees, have been so well attended during the past year.

Subcommittee I on Electrical Heating and Resistance Materials (C. W. Armstrong, chairman) submitted the proposed Standard Specifications for Drawn or Rolled Nickel-Chromium and Nickel-Chromium-Iron Alloys for Electrical Heating Elements, referred to earlier in the report. Approval of these specifications will make Specifications B 82 and B 83 obsolete; therefore the subcommittee is recommending their withdrawal.

---

[2] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 3.

[3] 1958 Book of ASTM Standards, Part 3.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The subcommittee also proposed the Tentative Method of Accelerated Life Test of Iron-Chromium-Aluminum Alloys for Electrical Heating referred to earlier in the report, and has submitted the revisions of the Method of Accelerated Life Test for Metallic Materials for Electrical Heating (B 76 – 57) listed earlier.

The subcommittee is currently revising the Tentative Specifications for High-Resistivity, Low - Temperature - Coefficient Wire (B 267 – 53 T).

Subcommittee II on Wrought and Cast Alloys for Structural Use at High Temperatures (W. A. Blonn, chairman) has been working on methods of testing high-temperature creep of structural materials and high-temperature corrosion. However, Subcommittee II will tentatively be inactive since Mr. Blonn has discontinued his membership in Committee B-4 because of a change in company duties and Mr. G. F. Geiger, Secretary, has discontinued his membership in Committee B-4 because of his retirement from the International Nickel Co.

Subcommittee III on Thermostat Metals (U. U. Savolainen, chairman) is considering test procedures which would cover such problems as flexivity, thermal emissivity, thermal conductivity, torque rate of bimetal coils, thermal expansion, and corrosion of bimetal materials.

Subcommittee IV on Contact Materials (J. D. Kleis, chairman) prepared the Tentative Method of Test for Surety of Make of Electrical Contact Materials (B 340 – 59 T). Revision of the standard tests for life test of electrical contact materials is being considered.

A list of over 300 terms in the study of nomenclature and definitions in the contact field has been prepared, while publication of the Bibliography and Abstracts on Electrical Contacts is continuing.

Work is continuing on measurement of physical properties of contact materials. Arc wear under high current conditions is being studied at currents up to 20,000 amp. Activity in the microcontact field is continuing, with forces as low as 0.1 g being considered. Atmospheric aging studies of connector and connection wire continue. Preliminary work is being done on evaluation of methods of test on sliding contacts of the instrument type.

This report has been submitted to letter ballot of the committee, which consists of 62 members; 55 members have returned their ballot, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

ERLE I. SHOBERT II,
Chairman.

C. K. STROBEL,
Secretary.

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT*

Committee B-5 on Copper and Copper Alloys held two meetings during the year: at Lake Placid, N. Y., in September, 1958, and at New York City in January, 1959.

The committee consists of 129 members of whom 124 are voting members; 54 are classified as producers, 55 as consumers, and 20 as general interest members.

G. R. Gohn has resigned as chairman of Subcommittee G-1 with P. D. Strubhar taking over the leadership. Mr. Sidney Metzger requested to be relieved of the chairmanship of Subcommittee W-3, and M. J. Mianulli was appointed as Chairman in his place.

The Supervisory Group of the committee continued to function as advisor to the American Standards Association in its representation of the United States as Secretariat of Technical Committee 26 on Copper and Copper Alloys of the International Organization for Standardization (ISO); L. H. Adam, A. E. Beck, R. S. Pratt, and J. S. Smart attended the June, 1958, meeting of ISO/TC-26 at Harrogate, England. Agreements were reached on Methods of Tests for Residual (Internal) Stresses of Copper and Copper-Base Alloys (ASTM Method B 154) and Method of Test for Expansion of Copper and Copper Alloy Tubing (ASTM Method B 153). Other items under study are draft specifications for electrolytic copper bar, cakes, slabs, billets, ingot and ingot bars as well as test methods related to the field of copper and copper alloys.

Research Activities.—Two technical papers were presented at committee meetings. At both the June and September meetings of Committee B-5, M. N. Torrey, G. R. Gohn, and M. B. Wilk, of the Bell Telephone Laboratories, presented "A Study of the Variability in the Mechanical Properties of Alloy A Phosphor Bronze Strip."[1] Mr. D. E. Trout, of Scovill Mfg. Co., presented his data on "Effect of Gage on Rockwell Hardness Testing of Brass Strip"[2] at the January meeting.

A task group consisting of W. F. Roeser (chairman), W. D. France, G. R. Gohn, and J. S. Roberts was assigned the project of reviewing the research projects under the jurisdiction of Committee B-5 for the Administrative Committee on Research.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee B-5 presented to the Society through the Administrative Committee on Standards the following recommendations:

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Published in *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 893 (1958).

[2] See p. 212.

Revision of Tentative Specifications for:

General Requirements for Wrought Copper and Copper-Alloy Wire (B 250 - 58 T), and

Rectangular Copper Wire for General Purposes (B 272 - 52 T).

These recommendations were accepted by the Standards Committee on September 9, 1958, and the revised tentative specifications are available as separate reprints, and they will appear in the 1959 Supplement to Book of ASTM Standards, Part 2.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is recommending the adoption as standard of seven tentatives, of which two are being revised. Revisions are proposed for immediate adoption in five standards. Tentative revisions of two standards are recommended.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee W-1 on Plate, Sheet and Strip (A. E. Moredock, chairman).—Tentative Specification B 122 and General Requirements B 248 are being recommended for adoption as standard. Specification B 122 was revised with respect to copper limits of alloy No. 8. Specification B 100 was revised with a clarification of Section 6(*a*). Task groups are working on new specifications for copper sheet sold by ounce weight (roofing copper); a new specification for copper foil; and a new specification for strip for flexible hose. Other task groups are considering schedules of close thickness tolerances for strip metals covered by Specification B 248, and revision of Specification B 100 to include mechanical properties for sizes not now covered by the specification. A task group is considering revision of the Rockwell hardness limits for various tempers and alloys in Specification B 36 as a result of thorough studies of correlation of hardness, tensile strength, and specimen thickness conducted by a task group in Subcommittee G-1.

Subcommittee W-2 on Rod, Bars, and Shapes (J. D. MacQueen, chairman).—Standard Specification B 16 was revised to include hard temper rod in sizes above $\frac{5}{16}$ in.; tentative Specifications B 249 and B 301 are being adopted as standard. Work on a new specification for leaded brass rod (1 to 2 per cent lead) is nearing completion. Revisions of Specifications B 16, B 187, B 140, and B 249 are being considered by task groups.

Subcommittee W-3 on Wire and Wire Rod (M. J. Mianulli, chairman) is studying the straightness tolerance in Specification B 250 and expanding the scope of Specification B 159 to include rectangular and square phosphor bronze wire.

Subcommittee W-4 on Pipe and Tube (G. C. Mutch, chairman) recommended revisions in Specifications B 75, B 188, B 251, B 302, and B 306, while Specifications B 251, B 280 and B 315 are to be adopted as standard. Task groups are working on surface oxidation of oxygen-free tube (and rod); dimensional tolerances for square and rectangular tube in Specifications B 75, B 135, and B 188; product specification for seamless rectangular wave guide tube; the addition of alloy "B" to Specification B 315; and specifications for integral finned tube and for capillary tube.

Other items under consideration by the committee are the establishment of

---

[3] See p. 209.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Specification B 88 as a self-contained product specification; revisions of the iron content of copper-nickel tubing in Specification B 111; revisions of Section 12(*d*) of Specification B 188; elimination from Specification B 280 of the requirement for microscopic examination of DHP copper for cuprous oxide; and a communication from G. A. Nelson representing a task group on residual stresses in condenser tubes, organized under Subcommittee IX of Committee A-1.

Subcommittee F-1 on Castings and Ingots for Remelting (G. H. Clamer, chairman) has appointed a Specification Review Task Group to revise all specifications under F-1 jurisdiction. This review is to bring all specifications to a uniform ASTM approved form and to correct minor discrepancies in chemical and physical requirements. Revisions of Specification B 30 are being recommended for immediate adoption. Revisions of standard Specifications B 61 and B 62 have been completed.

Specifications for investment castings; copper-nickel block, ingot and shot; and copper-nickel alloy castings are being prepared.

Subcommittee F-1 is cooperating with International Specification Group, ISO/TC 26 but is limiting its activities to correspondence. The casting group has decided not to send representation to the European meetings.

Subcommittee G-1 on Methods of Test (P. D. Strubhar, chairman).—It was determined that the ISO/TC 26 Mercurous Nitrate Test is compatible with ASTM Method B 154 without any change. Recommended revision of Table II in Specification B 36 has been completed.

Questionnaires from other ASTM committees were circulated to G-1 members and the following results reported: (1) The majority of the votes on the Shear and Torsion Questionnaire from Subcommittee 25 of Committee E-1 on Methods of Testing indicated no need for this test; and (2) the majority of the votes on the Grain Size Questionnaire from Subcommittee VIII of Committee E-4 on Metallography favored expanding the grain size charts to include 0.005-mm, 0.020-mm, 0.150-mm, and 0.200-mm grain sizes.

The following items are still under consideration by G-1: (1) compatibility of ISO/TC 26 (53E) Pin Test with the related ASTM specification; (2) compatibility of ISO/TC 26/WGI Mechanical Tests with the related ASTM specifications; and (3) a cylindrical correction table for hardness in Method E 18 requested by Subcommittee 6 of Committee E-1.

Subcommittee G-2 on Tolerances (J. E. McGraw, chairman).—At the request of Subcommittee W-4, subcommittee G-2 submitted appropriate dimensional tolerances for square and rectangular tubing. The report was accepted at the last meeting of the subcommittee and referred to a task group for implementation to standard Specifications B 75, B 135, B 188 and tentative Specification B 251. The subcommittee is also working on straightness tolerances for square and rectangular wire and straightness tolerances on condenser tube for Subcommittees W-3 and W-4 respectively.

Subcommittee G-3 on Editorial and Publications (W. F. Roeser, chairman) held two meetings and recommended a proposed outline for the Specification Writing Subcommittees of Committee B-5 to follow in the preparation of new specifications and the revision of existing specifications.

A task group has been set up in Subcommittee G-3 to review the research projects under the jurisdiction of Committee B-5 for the Administrative Committee on Research.

This report has been submitted to letter ballot of the committee, which consists of 129 members; 81 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. JENNINGS,
Chairman.

L. H. ADAM,
Secretary.

APPENDIX I

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix, recommendations are given affecting certain specifications, both standard and tentative, referred to earlier in the report. The specifications appear in their present form in the 1958 Book of ASTM Standards, Part 2.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

Standard Specification for Phosphor Bronze Plate, Sheet, Strip and Rolled Bar (B 103 – 55):

Section 1(a).—Revise the last sentence to read "Six alloys are covered, designated alloys A[3], A1, B, B1, C[3], and D."

Table I.—Add the following chemical requirements for alloy A1 strip:

Tin, per cent	4.2 to 5.8
Phosphorus, per cent	0.03 to 0.35
Iron, max, per cent	0.10
Lead, per cent	0.05 max
Zinc, max, per cent	0.30
Copper plus tin, plus phosphorus, min, per cent	99.5

Table II.—Add as a new section under the heading "Alloy A1 Strip" the requirements of the accompanying Table II.

Tentative Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 – 58 T):

In a subsequent section of this Appendix the committee is recommending the adoption as standard with revision of this specification. The following tentative revisions are also recommended for this specification:

Section 8.—To Paragraph (*a*) add the following as Item (*4*): "When tube is ordered by outside and inside diameters, the maximum plus and minus deviation of the wall thickness from the nominal at any point shall not exceed the values given in Table V by more than 50 per cent."

In Paragraph (*h*) add the following sentence to Item (*3*): "Tube in straight lengths shall be furnished in stock lengths with ends unless the order requires specific lengths or specific lengths with ends."

Table V.—Add the same statement as recommended above for Item (*4*) in Section 8(*a*), as a footnote to Table V.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip, and Rolled Bar (**B 248 – 58 T**),

General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (**B 249 – 58 T**),

Seamless Copper Tube for Refrigeration Field Service (**B 280 – 58 T**), Tellurium Copper Rod (B 301 – 58 T), and Copper-Silicon Alloy Seamless Pipe and Tube (B 315 – 58 T).

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Specification for Copper-Nickel-Zinc Alloy (Nickel Silver) and Copper-Nickel Alloy Plate, Sheet, Strip, and Rolled Bar (B 122 – 55 T):

Section 1.—In the nominal composition for alloy No. 8 change the copper content from the present "56.5" to read "59" per cent and change the zinc content from the present "31.5" to read "29" per cent.

Table I.—In the first column revise alloy No. 8 to read "(59.0 Cu, 12.0 Ni, 29.0 Zn)." In column 2 change the required range of copper content from the present "55.0 to 58.0" to read "57.0 to 61.0" per cent.

Table II.—Revise alloy No. 8 to read "(59.0 Cu, 12.0 Ni, 29.0 Zn)."

Tentative Specification for General Requirements for Copper and Copper-Alloy Pipe and Tube (B 251 – 58 T):

Table II.—For type M tube add the additional requirements in the accompanying Table I.

TABLE I.—ADDITIONS TO TABLE II OF SPECIFICATION B 251 – 58 T.

Standard Water Tube Size, in.	Wall Thickness, in.		Theoretical Weight, lb per ft
	Nominal	Tolerance	
⅜	0.025	0.0025	0.145
½	0.028	0.0025	0.204
¾	0.032	0.003	0.328
1	0.035	0.0035	0.465

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society:

Standard Specification for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 54):

This specification was extensively rearranged and revised. The completely revised document is appended.[1]

Standard Specification for Seamless Copper Tube (B 75 – 58):

Section 11.—Revise the tabular material and Note 3 to read as follows:

Condition	Electrical Resistivity, max, ohm-gram/meter^2	
	OF Copper	DLP Copper
Annealed tempers	0.15328	0.15614
Drawn tempers	0.15737	0.15940

NOTE 3.—The International Annealed Copper Standard electrical conductivity equivalents are as follows:

Electrical Resistivity, ohm-gram/meter^2	Conductivity per cent
0.15328	100.00
0.15614	98.16
0.15737	97.40
0.15940	96.16

Standard Specifications for Seamless Copper Bus Pipe and Tube (B 188 – 58):

Table IV.—For Type M add additional requirements as indicated above for Table II of Specification B 251.

Standard Specification for Threadless Copper Pipe (B 302 – 58):

Section 13(d).—Revise to read "*Roundness Tolerance.*—The roundness tolerances shall be in accordance with Table II."

---

[1] The revised standard appears in the 1959 Supplement to Book of ASTM Standards, Part 2.

TABLE II.—ADDITIONS TO TABLE II OF SPECIFICATION B 103 - 55.

Temper	Thickness, in.	Tensile Strength, psi		Approximate Rockwell Hardness					
				B Scale		G Scale		30-T Scale	
		Min	Max	Min	Max	Min	Max	Min	Max
ALLOY A1 STRIP									
Soft	Over 0.039	43 000	58 000	16	64	..	28	..	..
	Over 0.029			..	..	..	..	32	59
	0.020 to 0.039, incl			12	60	..	..	..	..
	0.010 to 0.029, incl			..	..	..	..	24	53
Half-hard	Over 0.039	58 000	73 000	64	85	28	56	..	..
	Over 0.029			..	..	..	..	59	73
	0.020 to 0.039, incl			60	82	25	54	..	..
	0.010 to 0.029, incl			..	..	..	..	53	69
Hard	Over 0.039	76 000	91 000	86	93	58	69	..	..
	Over 0.029			..	..	..	..	73	78
	0.020 to 0.039, incl			84	91	56	67	..	..
	0.010 to 0.029, incl			..	..	..	..	71	75
Extra-hard	Over 0.039	88 000	103 000	92	96	67	75		
	Over 0.029			..	..	..	..	77	81
	0.020 to 0.039, incl			89	95	65	73	..	..
	0.010 to 0.029, incl			..	..	..	..	74	78
Spring	Over 0.039	95 000	110 000	94	98	71	78	..	..
	Over 0.029			..	..	..	..	79	82
	0.020 to 0.039, incl			92	97	69	76	..	..
	0.010 to 0.029, incl			..	..	..	..	76	80
Extra-spring	Over 0.039	100 000	114 000	95	99	73	80	..	..
	Over 0.029			..	..	..	..	80	83
	0.020 to 0.039, incl			94	98	71	78	..	..
	0.010 to 0.029, incl			..	..	..	..	77	81

New Table.—Add a new Table II to read as shown in the accompanying Table III.

Standard Specification for Copper Drainage Tube (DWV) (B 306 - 58):

Section 11(d).—Change to read as follows:

(*d*) *Roundness Tolerance.*—The difference between the major and minor outside diameters as determined at any one cross-section of the tube shall not exceed 1½ per cent, expressed to the nearest 0.001 in., of the actual outside diameter of the tube.

TABLE III.—ROUNDNESS TOLERANCES.

t/D, Ratio of Nominal Wall Thickness to Nominal Outside Diameter	Roundness Tolerances,[a] per cent of Nominal Outside Diameter (Expressed to the Nearest 0.001 in.)
0.01 to 0.03, incl	1.5
Over 0.03 to 0.05, incl	1.0
Over 0.05 to 0.10, incl	0.8
Over 0.10	0.7

[a] The deviation from roundness is measured as the difference between major and minor outside diameters, as determined at any one cross-section of the tube.

APPENDIX II

EFFECT OF GAGE ON ROCKWELL HARDNESS TESTING OF BRASS STRIP

By Delmar E. Trout[1]

Synopsis

The minimum thickness of strip that can possibly be tested safely for Rockwell hardness has long been controversial. For more than a decade, ASTM Committee B-5 on Copper and Copper Alloys felt that the minimum strip thickness should be increased to ensure hardness measurements undistorted by anvil effect. In the past, several manufacturers undertook production tests to obtain a definite answer to this question. Although these tests indicated to some extent an effect of specimen thickness on the measured hardness values, large scatter of the plotted data rendered the results inconclusive. In an attempt to minimize this scatter, the Scovill Manufacturing Co. has recently completed studies on brass strip, designed to investigate more comprehensively this effect of specimen thickness on Rockwell hardness measurements. This paper reviews and interprets the tabulated test results and makes recommendations for changes in strip specifications.

Presently, most ASTM specifications for copper and copper alloy strip, cast and wrought, include Rockwell hardness values as information only, not as requirements. These hardness values correspond to the minimum and maximum tensile strength values for rolled tempers. In these specifications, footnotes to the tables of tensile strength and hardness values generally state the minimum strip thickness which may be tested with each hardness scale indicated. For example, the Specification for Brass Plate, Sheet, Strip, and Rolled Bar (B 36),[2] the primary specification to be discussed here, indicates a minimum thickness of 0.020 in. for the Rockwell B scale and 0.012 in. for the Rockwell 30-T superficial hardness scale.

About 13 years ago, Subcommittee G-1 on Methods of Test of ASTM Committee B-5 on Copper and Copper Alloys investigated the question of minimum thickness for Rockwell hardness testing of strip in specifications under the jurisdiction of Subcommittee W-1 on Plate, Sheet, and Strip. It was felt that, in specifications originating from Subcommittee W-1, the minimum thickness should be increased to ensure hardness values free from anvil effect and more representative of the corresponding tensile strength. Based on a method published by Peck and Ingerson,[3] a table of minimum thickness grades for different hardness values and scales was referred by Committee B-5 to ASTM Committee E-1 on Methods of Testing for study. It was found that these thickness values were not acceptable to producer members. Little progress was made toward a solution of this assignment until results of research work were reported by Gohn et al[4] which again indicated the effect of gage thickness on the hardness of phosphor bronze strip, the hardness values for the range between 0.020 and 0.036 in. gage thickness being 1 to 3 points less on the Rockwell B scale than for 0.040 to 0.063 in. strip of the same tensile strength.

---

[1] Research Metallurgist, Scovill Manufacturing Co., Waterbury, Conn.

[2] 1958 Book of ASTM Standards, Part 2.

[3] R. L. Peck, Jr., and W. E. Ingerson, "Analysis of Rockwell Hardness Data," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 1270 (1939).

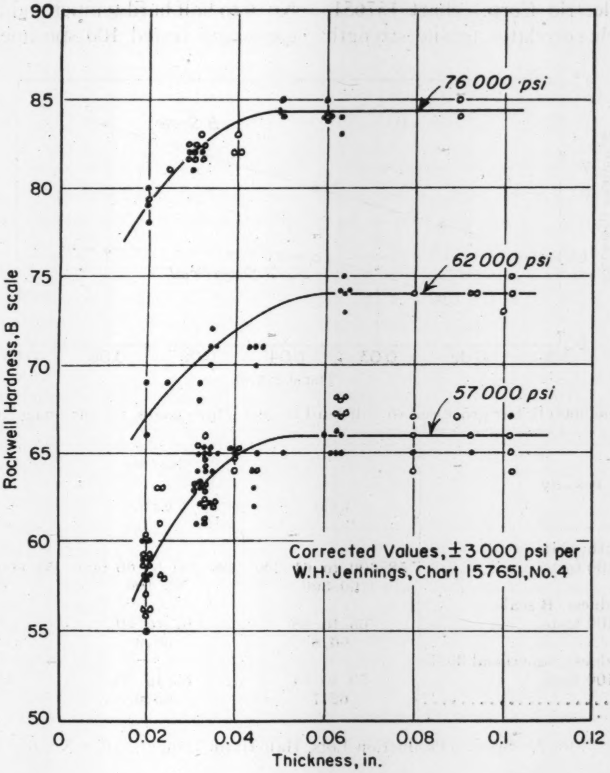
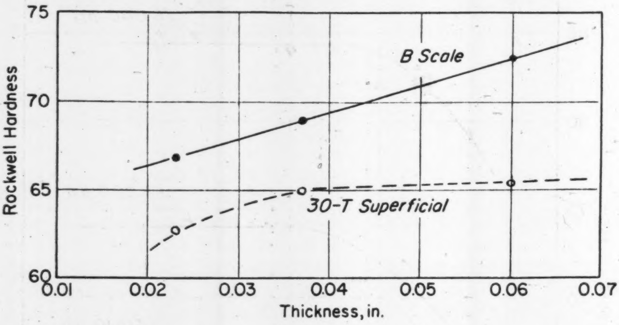


FIG. 1.—Rockwell Hardness, B Scale, *Versus* Gage for Constant Tensile Strength, Alloy No. 6 (Scovill 1956 Production Test).

Western Electric Corp., Chase Brass and Copper Co., and the Scovill Manufacturing Co. submitted to Subcommittee G-1 data taken from routine production tests which showed a great similarity relative to the effect of gage thickness. Figure 1, a chart of Scovill 1956 production test results, shows as an example the relationship of gage *versus* Rockwell B hardness for alloy No. 6 of Specification B 36 at three constant tensile strength values. Many of these values are corrected, ±3000 psi according to Jennings (Western Electric Corp.) chart 157651, No. 4, which correlates tensile strength to Rockwell B hardness of alloy No. 6. This chart shows that, with constant tensile strength, the Rockwell B hardness increases from 0.020 in. to 0.050 or 0.060 in. There is apparently no effect due to gage above 0.050 or 0.060 in. Although these production tests indicate an effect of gage, the data are inconclusive because of the scatter of hardness values, especially at the lower tensile strength values. The large spread may be due to variables such as chemical analyses, grain size effects, surface finish, and errors in correcting for temper.

---

[4] G. R. Gohn, J. P. Gerrard, and E. S. Freynik, "The Mechanical Properties of Wrought Phosphor Bronze Alloys," Am. Soc. Testing Mats. (1956). (Issued as a separate publication *ASTM STP No. 183*.)



NOTE.—One 3000-lb bar processed to half-hard temper, three gages, for each gage 100 specimens tested.

Property	Gage, in.		
	0.023	0.037	0.060
Tensile strength, psi:			
Spread of 100 tests	58 600 to 61 200	58 300 to 60 900	59 600 to 61 600
Mean	60 300	60 100	60 700
Rockwell hardness, B scale:			
Spread of 100 tests	65 to 68	67 to 70	71 to 73
Mean	66.8	68.9	72.4
Rockwell hardness, superficial 30-T:			
Spread of 100 tests	59 to 64	63 to 66	63 to 66
Mean	62.7	65.0	65.4

FIG. 2.—Special Production Lots, Half-Hard Temper, Alloy No. 6.

To minimize the spread of the values noted in production test compilations, Scovill Manufacturing Co. processed one 3000-lb bar of specification B 36 alloy No. 6 to half hard temper and three finish gages and tested 100 specimens of each of the three gages. The results are summarized in Fig. 2. The spread and the mean tensile strength values for each gage are similar, but notable differences in mean hardness values were found for both the Rockwell B scale and the 30-T superficial hardness scale. This effect is plotted in Fig. 2. The results obtained with these special test lots indicate that the slope of the production data is real and the hardness at constant tensile strength is a function of gage.

A special study designed to investigate in a more comprehensive manner this effect of gage on the Rockwell hardness test of brass strip has been completed by the Scovill Manufacturing Co. This paper reviews the tabulated results, gives an interpretation of these results, and makes suggestions and recommendations which might be considered by Committee B-5 for strip specifications.

Test Procedures

This special study concerns alloys Nos. 1, 3, and 6 of Specification B 36, processed to finish gage sizes between 0.018 and 0.072 in. for tempers of eighth hard to extra spring from two ready-to-finish annealed tempers. For each alloy there were approximately 200 test conditions. The procedure for deriving the test results is as follows:

1. All specimens were processed from a single 3000-lb bar, selected at an intermediate rolling gage of 0.460 to 0.560 in., depending on the alloy, subsequently cold rolled, annealed, and tested in the Metals Research Department of the Scovill Manufacturing Co.

2. The chemical composition of the individual bars is given below:

	Alloy No. 1 (Gilding, 95 per cent)	Alloy No. 3 (Commercial Bronze, 90 per cent)	Alloy No. 6 (Cartridge Brass, 70 per cent)
Copper, per cent.....	94.64	84.85	70.41
Lead, per cent.....	0.01	0.01	0.02
Iron, per cent.....	0.01	0.01	0.02
Zinc, per cent.....	remainder	remainder	remainder

3. Annealing was performed in a Lindberg recirculating furnace. The nominal ready-to-finish grain sizes were 0.015 mm for one half of the specimens and 0.050 mm for the other half. The grain sizes were checked according to the Methods for Estimating the Average Grain Size of Wrought Copper and Copper-Base Alloys (E 79).[5] Pre-ready anneals were adjusted accordingly to produce commercial microstructural uniformity.

4. Rolling was performed on a Fenn combination rolling mill (2 Hi–4 Hi) at the Scovill laboratory.

5. An attempt was made to finish to each of the rolled tempers of Specification B 36, plus an eighth hard temper, from both 0.015 and 0.050-mm annealed tempers, for Brown & Sharpe gage Nos. 25 to 13 or 0.018 to 0.072 in., inclusive. The exact finish gages, 0.016 to 0.078 in., were used in plotting the test data.

6. The hardness values and the mechanical properties such as tensile strength, yield strength, and elongation were determined according to the Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18)[5] and the Methods of Tension Testing of Metallic Materials (E 8),[5] respectively.

Test Results and Interpretation of Data

For each alloy and for both the Rockwell B and 30-T hardness values, plots of hardness *versus* thickness were made for each rolled temper. Figure 3 shows the plot for Rockwell B scale hardness for Alloy No. 6. There is a scatter of hardness results for the half hard and quarter hard tempers, especially for the quarter hard specimens. As the temper increases, this scatter apparently decreases, and a clearer definition of the effect of gage is noted. The Rockwell B scale data for all alloys were similar with regard to scatter for tempers and gage effects. The data for all alloys fall within the listed hardness ranges within statistical expectations. This condition indicates the possibility of maintaining the present total over-all Rockwell B hardness range for each temper of Specification B 36 and tightening the hardness ranges for different gage ranges. A study of the Rockwell B scale plots for alloys Nos. 1, 3, and 6 reveals that 0.036 in. would be a good gage break point (corresponding with the standard Brown & Sharpe gage No. 19). The data did not indicate a necessity for more than two gage breaks.

---

[5] 1958 Book of ASTM Standards, Part 3.

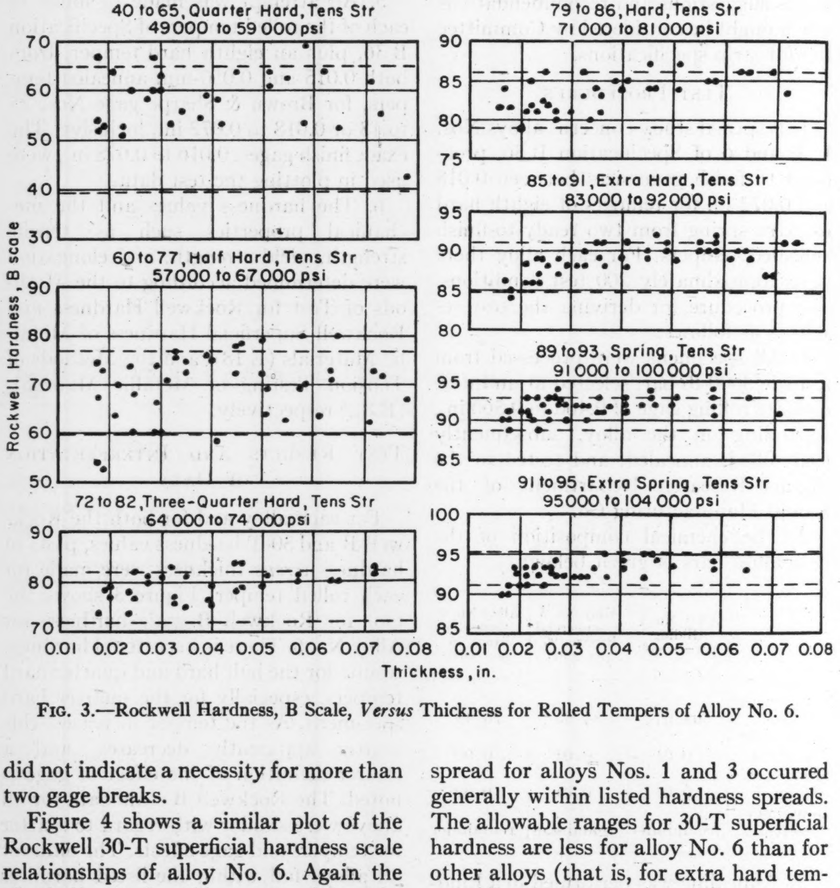


FIG. 3.—Rockwell Hardness, B Scale, *Versus* Thickness for Rolled Tempers of Alloy No. 6.

Figure 4 shows a similar plot of the Rockwell 30-T superficial hardness scale relationships of alloy No. 6. Again the plot indicates the wide scatter of the soft-rolled tempers as compared to the harder tempers and a similar effect due to gage. This scatter and gage effect is similar for all three alloys. The data again indicate that only two gage breaks are necessary with 0.028 in. as the best gage break corresponding with Brown & Sharpe gage No. 21. However, the 30-T superficial hardness values for alloy No. 6 did not fall within the listed hardness range within statistical expectations. The spread for alloys Nos. 1 and 3 occurred generally within listed hardness spreads. The allowable ranges for 30-T superficial hardness are less for alloy No. 6 than for other alloys (that is, for extra hard temper: alloy No. 1, 5 points; alloy No. 3, 5 points; and alloy No. 6, 3 points. Although the total spread for 30-T superficial hardness of alloy Nos. 1 and 3 occurred within the permitted spread, the range was generally 1 to 3 points higher than the range specified in Specification B 36. In actual production, the present limits of the Rockwell B and 30-T scale values called for in Specification B 36 have been found satisfactory with the exception of the 30-T scale values for alloys Nos. 1, 2, and 3 for which test values seem to be higher than those listed. This condition has been verified by the test results under consideration.

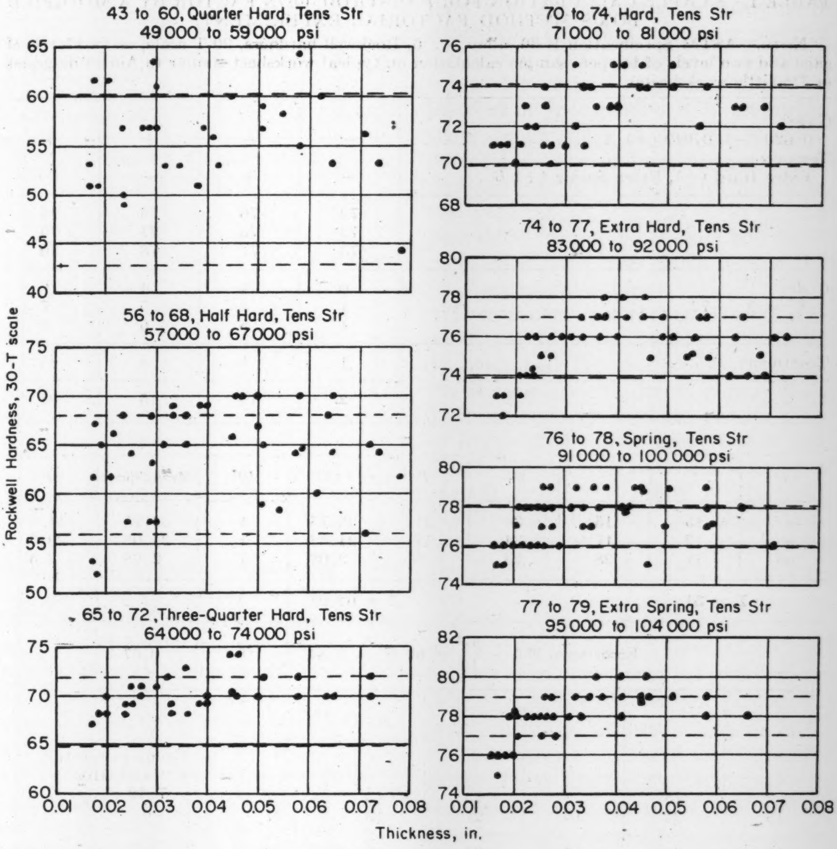


FIG. 4.—Rockwell Hardness, 30-T Scale, *Versus* Thickness for Rolled Tempers of Alloy No. 6.

To determine whether the visual assumptions of two thickness ranges and the exact gage breaks selected are logical statistically, the data were subjected to a mathematical analysis of variance using a modified Box factorial experiment at two levels of gage and two levels of temper. By determination of the *F* distribution factor, it should be possible to establish whether the data for two different gages are from similar populations and, if not, to find the degree of significance of the difference. Table I illustrates a sample calculation for *F* distribution. Table II summarizes some of the *F* distribution factors determined. The lower portion of Table II shows *F* distribution factors at various percentage fiducial levels. Industry generally accepts as significant the 0.05 level or 95 chances in 100 that there is a real difference, and as very significant the 0.01 level or 99 chances in 100. At levels 0.01 or lower there is no doubt that the differences denoted by *F* ratios are significant. Table II(*a*) shows no significance between 0.020 in. *versus* 0.030 in. nor between 0.040 in. *versus* 0.060 in. There is very strong and similar significance between 0.020 in. *versus* 0.040 in. and between 0.020 in. *versus* 0.060 in. The difference between 0.030 in. *versus* 0.040 in. is very significant. Table II(*a*) seems to indicate that only two gage ranges are significant and that the break

TABLE I.—SAMPLE CALCULATION FOR *F* DISTRIBUTION FACTOR BY A MODIFIED BOX'S METHOD FACTORIAL EXPERIMENT.

NOTE.—ASTM Specification B 36, alloy No. 6, Rockwell hardness, 30-T scale, at two levels of gage and two levels of temper (sample calculation on typical worksheet similar to Anova diagrams or Doolittle worksheets).

Gage: 0.020 (−), 0.040 (+) *A*	−	−	+	+
Temper: Extra Hard (−), Extra Spring (+) *B*	−	+	−	+
	72 73 73	76 76 77	76 77 78	78 79 80
Code: $X' = X - 72$	0 1 1	4 4 5	4 5 6	6 7 8
Treatment	1	*b*	*a*	*ab*
Σ	2	13	15	21

1	2	Σ−	Σ+	*T*	$T^2/12$	*DF*	Mean Square	*F*
a	15	15	36	21	36.75	1	36.75	44.8
b	13	17	34	17	24.08	1	24.08	29.5
ab	21	28	23	5	2.08	1	2.08	2.5
Σ = 51					Σ = 62.91	3	....	....
Error term $285 - \frac{51^2}{12} - 62.91 = 5.34$						8	0.67	....

a		*b*		*ab*		
−	+	−	+	−	+	Group interaction and error $\frac{7.42}{9} = 0.82$
2 13	15 21	2 15	13 21	13 15	2 21	
15	36	17	34	28	23	

TABLE II.—SUMMARY OF *F* DISTRIBUTION FACTORS.

(*a*) Various Gage Combinations for Alloy No. 6, Rockwell B, Half Hard *versus* Extra Hard.

Gage Combinations, in.	*F*
0.020 *versus* 0.030	0.8
0.030 *versus* 0.040	11.3
0.040 *versus* 0.060	0.5
0.020 *versus* 0.040	23.9
0.020 *versus* 0.060	23.4

(*b*) Gage 0.020 *versus* 0.040 in.

Alloy and Scale	Half Hard *versus* Extra Hard	Extra Hard *versus* Extra Spring
No. 1, B	56.8	15.3
No. 1, 30-T	34.4	18.6
No. 3, B	17.0	11.0
No. 3, 30-T	19.3	29.8
No. 6, B	23.9	3.6
No. 6, 30-T	16.0	44.8

For $n_1 = 1, n_2 = 9$

P	0.5	0.1	0.05	0.025	0.01	0.005	0.001
F	0.494	3.36	5.12	7.21	10.6	13.6	22.9

should be made between 0.030 and 0.040 in. Similar relationships were found for 30-T superficial hardness values which, when tested in this manner, suggest two gage ranges with the break either at 0.025 in. or at 0.030 in. Table II(*b*) shows *F* ratios of the three alloys for 0.020 in. *versus* 0.040 in. on both B scale and 30-T superficial hardness scale at two pairs of temper levels. Of the 12 mathematical analyses listed, 11 indicate great significance or less than 1 in 100 chances that there is not a real difference. On the B scale, extra hard *versus* extra spring hardness, alloy No. 6 had an *F* distribution factor of 3.6 or approximately 1 in 10 chances of significance which is a probable, not a positive level. This special study was not planned to be analyzed subsequently mathematically. However, the fact is most positive that 11 of the 12 tests were very significant. If this had been a planned experiment, it is very likely that, at the 0.1 *F* ratio, the twelfth sample would have been very significant.

Based on the visual study of the plotted data and the mathematical analysis, it seems apparent that gage is a positive factor and that a revision of hardness values for various gage sizes might be justified in Specification B 36 and other strip specifications originating in Committee B-5. The findings prompted further analysis of the data with regard to two gage ranges, 0.020 to 0.036 in. and over 0.036 in. for the B scale and 0.012 to 0.028 in. and over 0.028 in. for the 30-T superficial hardness scale.

The selector chart (Table II of Methods E 18)[5] for scales based on the $\frac{1}{16}$-in. diameter ball indenter shows that, for the B scale, any thickness greater than 0.036 in. and any hardness greater than 40 Rockwell B can be safely tested. For the 30-T superficial hardness scale, this selector chart relationship is 34 Rockwell 30-T and 0.024 in. Both values correlate quite well with the gage breaks found best in the tests under consideration.

Figures 5 to 10 show hardness *versus* tensile strength for the three alloys and the two gage ranges selected with a clear indication of the differential. Figures 11 to 13 illustrate Rockwell B *versus* 30-T relationships for the three alloys investigated. These plots show only tests for gage sizes over 0.036 in., which are not considered significant for either scale. Figure 11 concerns alloy No. 6, Specification B 36, and shows good correlation with both Table V of the Hardness Conversion Tables for Metals (E 140)[2] and Wilson 52 conversion charts which are based on alloy No. 6 conversion test data. Figure 12 shows conversion data for alloy No. 3, Specification B 36. Besides the relationship between Table V

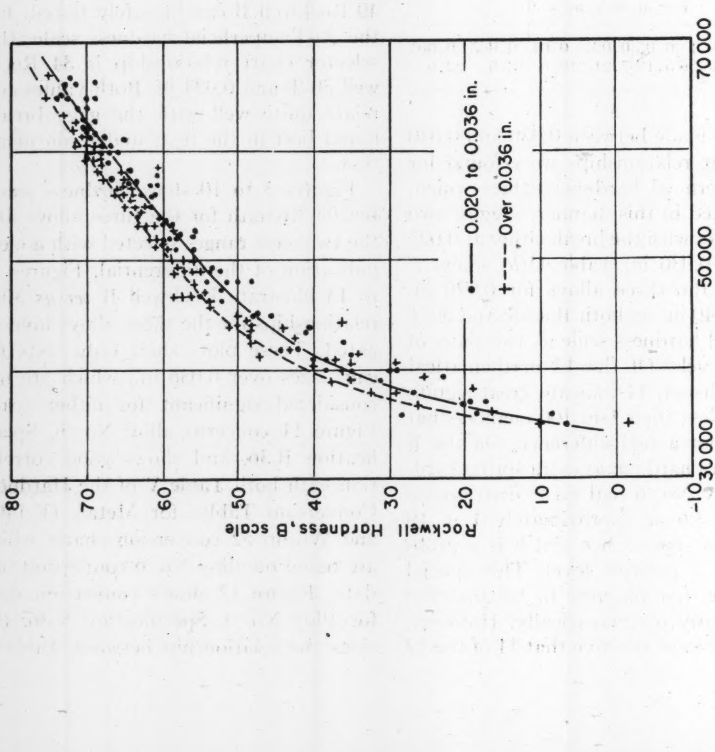


FIG. 5.—Rockwell Hardness, B Scale, *Versus* Tensile Strength for Alloy No. 1.

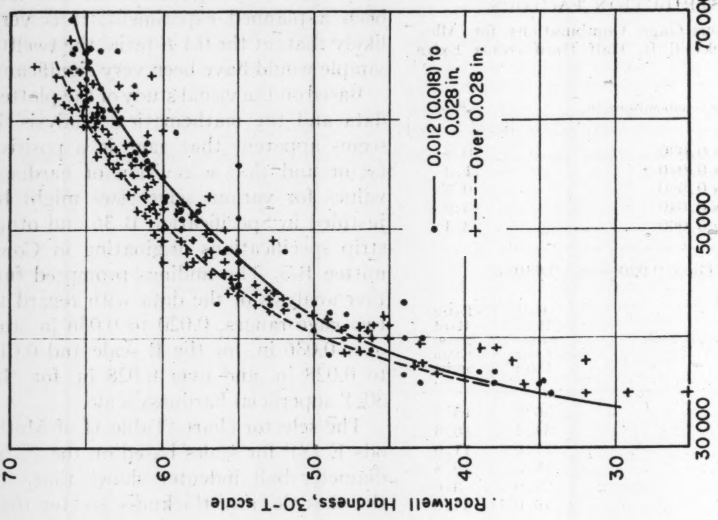


FIG. 6.—Rockwell Superficial Hardness, 30-T Scale, *Versus* Tensile Strength for Alloy No. 1.

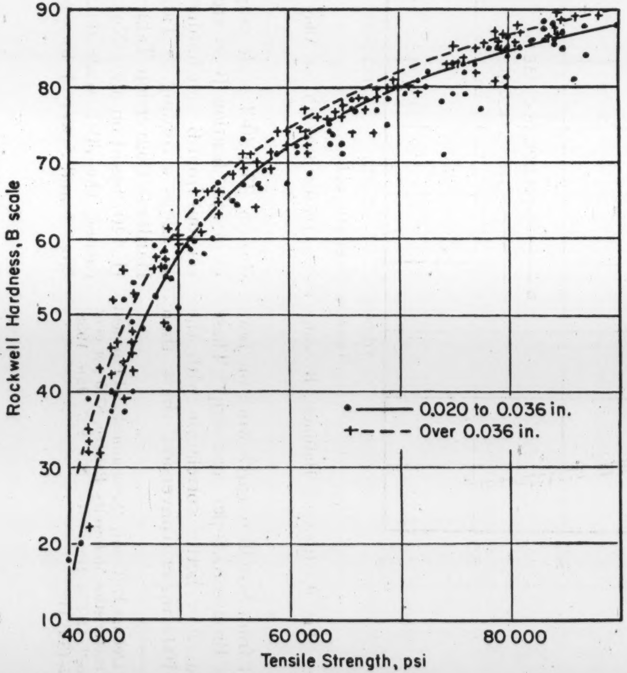


FIG. 7.—Rockwell Hardness, B Scale, *Versus* Tensile Strength for Alloy No. 3.

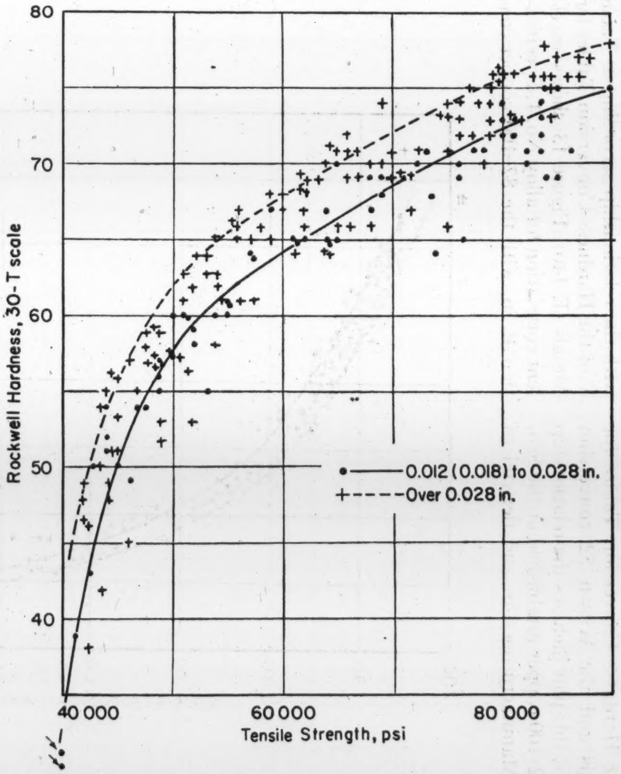


FIG. 8.—Rockwell Superficial Hardness, 30-T Scale, *Versus* Tensile Strength for Alloy No. 3.

of the Hardness Conversion Tables for Metals and the Wilson 52 conversion charts, this plot includes the relationship for 82–100 copper established at the Scovill Manufacturing Co. and derived in part from Scovill research work on Jewelry Bronze 87.5 per cent strip.[6] These data show better correlation with the 82–100 copper conversion chart than with the relationship according to Table V of the Hardness Conversion Tables for Metals (E 140). Figure 13 shows excellent correlation for alloy No. 1, Specification B 36, with the 82–100 copper conversion. The 30-T scale ranges in Table II of Specification B 36 are direct conversions from B scale hardness values for all alloys according to Table V in the Hardness Conversion Tables for Metals (E 140) based on alloy No. 6. As stated earlier, the 30-T scale data for alloys Nos. 1 and 3 were 1 to 3 points higher

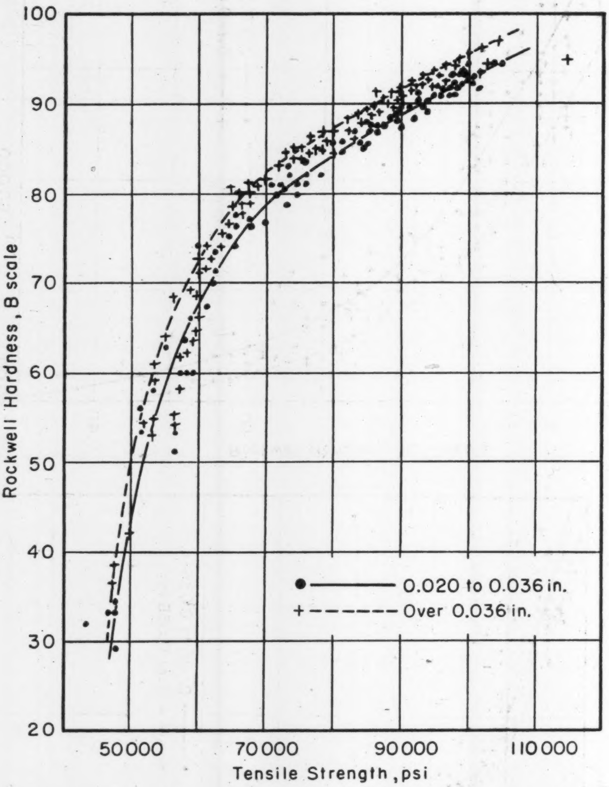


FIG. 9.—Rockwell Hardness, B Scale, *Versus* Tensile Strength for Alloy No. 6.

---

[6] Delmar E. Trout, "Mechanical and Physical Characteristics of Jewelry Bronze, 87.5 per cent Strip," ASTM BULLETIN, No. 227, Jan. 1958, p. 45 (TP 11).

than specified in Table II. Inaccurate conversion from the B scale to the 30-T scale is the obvious explanation for this discrepancy. For example, Table II lists for alloy No. 1, hard temper, 57 to 67 on the B scale and 54 to 62 on the 30-T scale. According to Table V of the Hardness Conversion Tables for Metals (E 140), 57 on the B scale corresponds with 54 on the 30-T scale and 67 on the B scale with 62 on the 30-T scale. According to Scovill's 82–100 copper chart, 57 on the B scale corresponds with 57 on the 30-T scale and 67 on the B scale with 64 on the 30-T scale. Except for two among 61 tests for alloy No. 1, the 30-T scale results at Scovill were between 57 and 64 and verified that conversion with the 82–100 copper chart is more realistic than with Table V of the Hardness Conversion Tables for Metals (E 140).

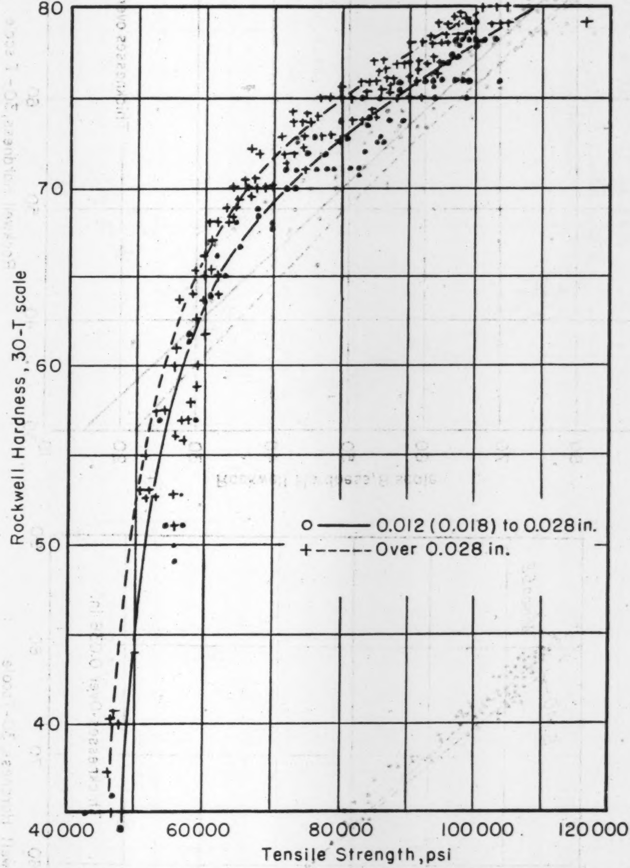


FIG. 10.—Rockwell Superficial Hardness, 30-T Scale, *Versus* Tensile Strength for Alloy No. 6.

As stated earlier, two ready-to-finish

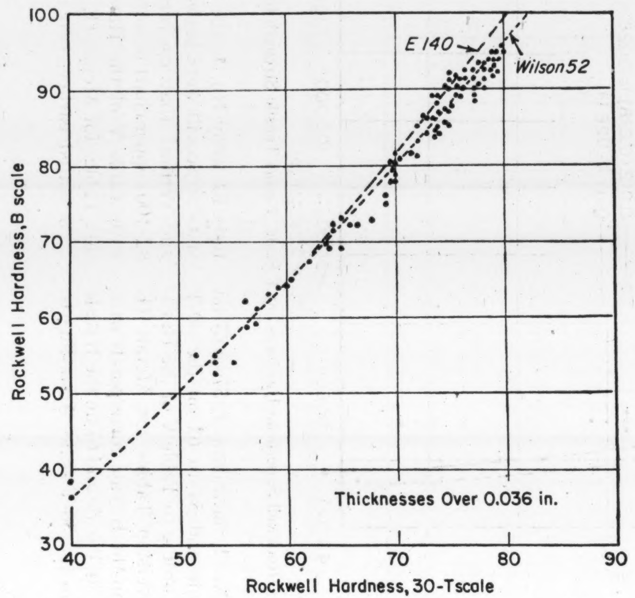


FIG. 11.—Rockwell Hardness, B Scale, *Versus* 30-T Scale for Alloy No. 6.

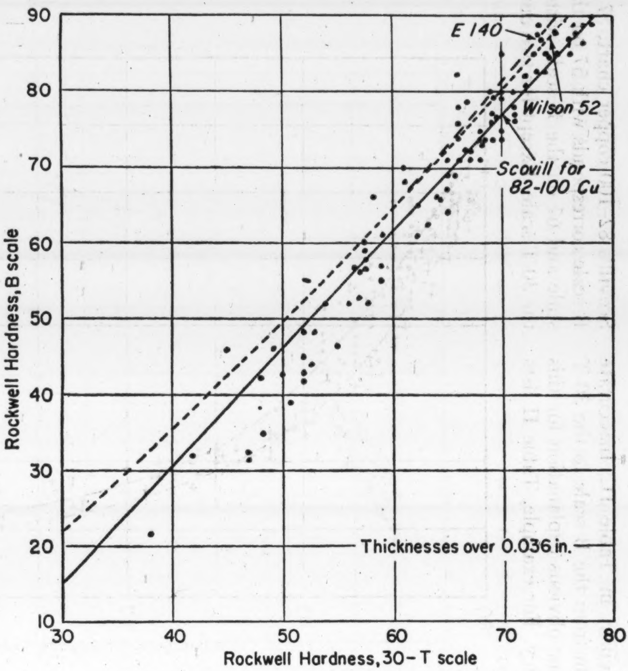


FIG. 12.—Rockwell Hardness, B Scale, *Versus* 30-T Scale for Alloy No. 3.

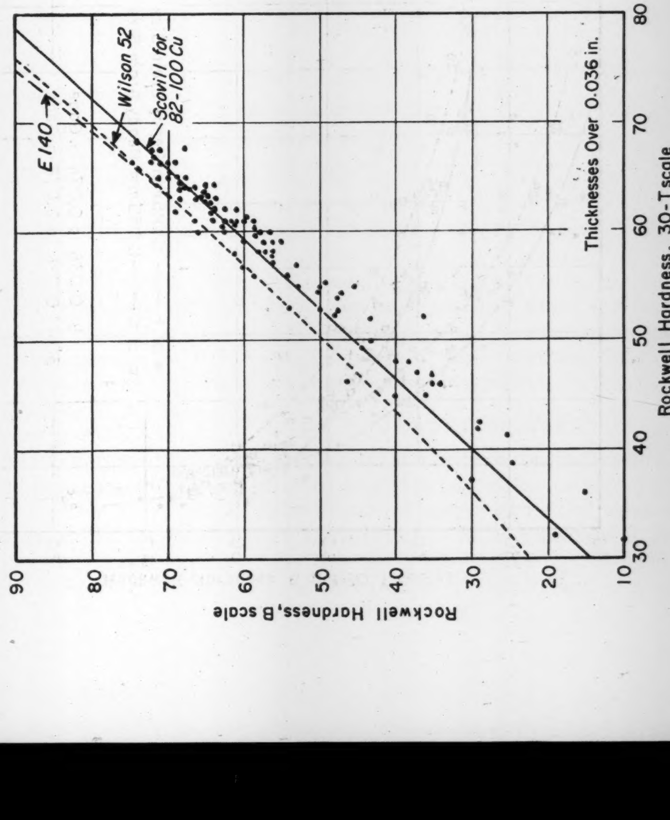


FIG. 13.—Rockwell Hardness, B Scale, *Versus* 30-T Scale for Alloy No. 1.

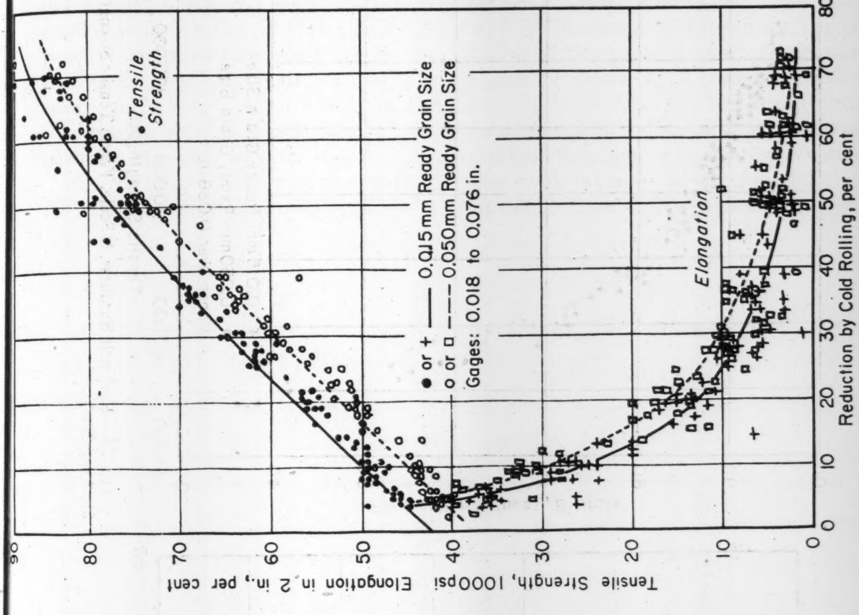


FIG. 14.—Effect of Cold Rolling on Tensile Strength and Elongation of Alloy No. 3.

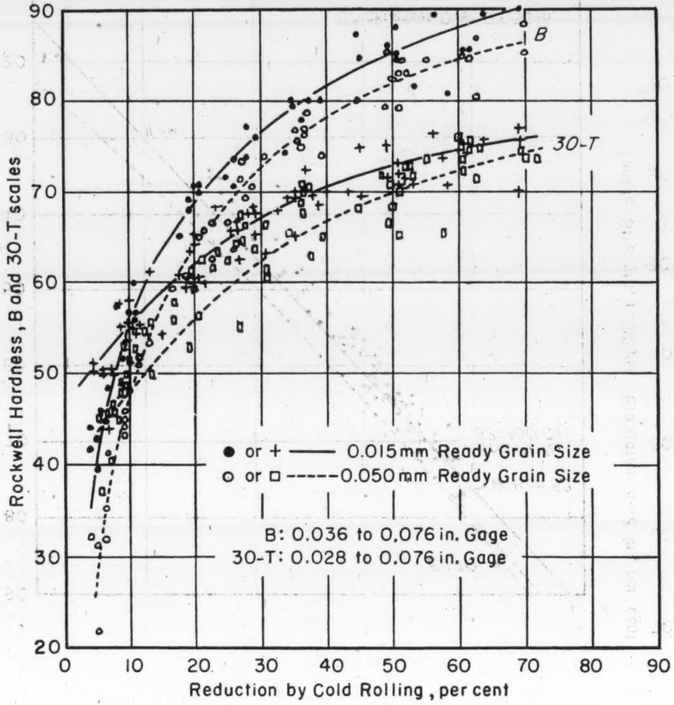


FIG. 15.—Effect of Cold Rolling on Hardness of Alloy No. 3.

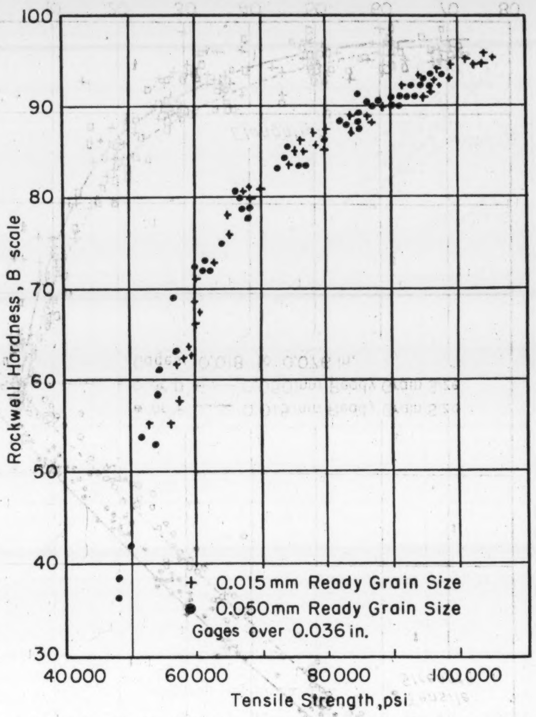


FIG. 16.—Rockwell Hardness, B Scale, *Versus* Tensile Strength for Alloy No. 6.

grain sizes, 0.015 and 0.050 mm, were employed in the Scovill tests. The purpose of this procedure was to verify the experience that, for plain brasses, the ready grain size has no effect on the relationship between tensile strength and hardness of rolled tempers.

Figures 14 to 17 illustrate the effect of the ready-to-finish grain size relative to this concept. Figure 14 shows the effect of cold rolling on the tensile strength and elongation at the two ready grain sizes of alloy No. 3, ASTM Specification B 36. The net result or displacement of properties due to the differential in the ready grain size is as anticipated. Figure 15 is similar to Fig. 14 but concerns Rockwell B and 30-T scale hardness. Similar relationships were found for alloys Nos. 1 and 3. Figure 16 shows the Rockwell B scale *versus* tensile strength relationships for alloy No. 6, ASTM Specification B 36, for the two ready grain sizes. The scatter of points is normal and denotes no interaction of the ready-to-finish grain size. Figure 17 shows the B scale *versus* 30-T scale correlation at the two ready grain sizes. Again this distribution is random and no interaction or correlation is noted. The same randomness was found for alloys Nos. 1 and 3. No evidence could be detected in the work under consideration that the ready-to-finish grain size has any correlation with the relationship of tensile strength *versus* hardness for the same gages or gage ranges.

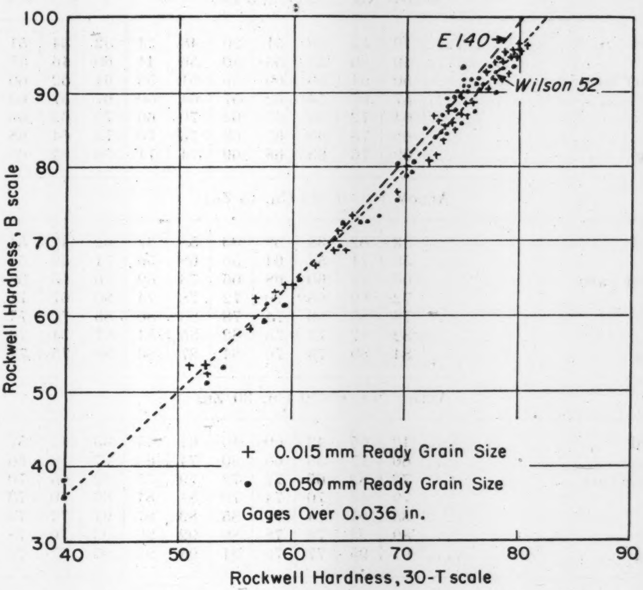


FIG. 17.—Rockwell Hardness, B Scale, *Versus* 30-T Scale for Alloy No. 6.

RECOMMENDATIONS

Table III summarizes the findings presented here in a plot of the present version *versus* the suggested revised version of Table II in ASTM Specification B 36 for the three alloys investigated. The over-all hardness spreads for any alloy or

TABLE III.—PRESENT *VERSUS* SUGGESTED REVISION OF TABLE II OF ASTM SPECIFICATION B 36.

Rolled Temper	Present				Suggested Revision							
	B		30-T		B				30-T			
	0.020 in. and Over		0.012 in. and Over		0.020 in. to 0.036 in.		Over 0.036 in.		0.012 in. to 0.028 in.		Over 0.028 in.	
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
ALLOY NO. 1 (95 Cu, 5 Zn)												
Quarter hard	20	52	29	51	20	48	24	52	34	51	37	54
Half hard	40	60	43	56	40	56	44	60	46	57	48	59
Three-quarter hard	50	64	50	60	50	61	53	64	52	60	54	62
Hard	57	67	54	62	57	64	60	67	57	62	59	64
Extra hard	64	72	60	65	64	70	66	72	62	66	63	67
Spring	68	75	62	67	68	73	70	75	64	68	65	69
Extra spring	69	76	63	68	69	74	71	76	65	69	66	70
ALLOY NO. 3 (85 Cu, 15 Zn)												
Quarter hard	33	62	38	58	33	58	37	62	42	57	45	60
Half hard	56	71	54	64	56	68	59	71	56	64	58	66
Three-quarter hard	66	76	60	68	66	73	69	76	63	68	65	70
Hard	72	80	65	70	72	78	74	80	67	71	68	72
Extra hard	78	85	69	74	78	83	80	85	70	74	71	75
Spring	82	87	72	75	82	85	84	87	74	76	75	77
Extra spring	84	89	73	76	84	87	86	89	75	77	76	78
ALLOY NO. 6 (70 Cu, 30 Zn)												
Quarter hard	40	65	43	60	40	61	44	65	43	57	46	60
Half hard	60	77	56	68	60	74	63	77	56	66	58	68
Three-quarter hard	72	82	65	72	72	79	75	82	65	70	67	72
Hard	79	86	70	74	79	84	81	86	70	73	71	74
Extra hard	85	91	74	77	85	89	87	91	74	76	75	77
Spring	89	93	76	78	89	92	90	93	76	78	76	78
Extra spring	91	95	77	79	91	94	92	95	77	79	77	79

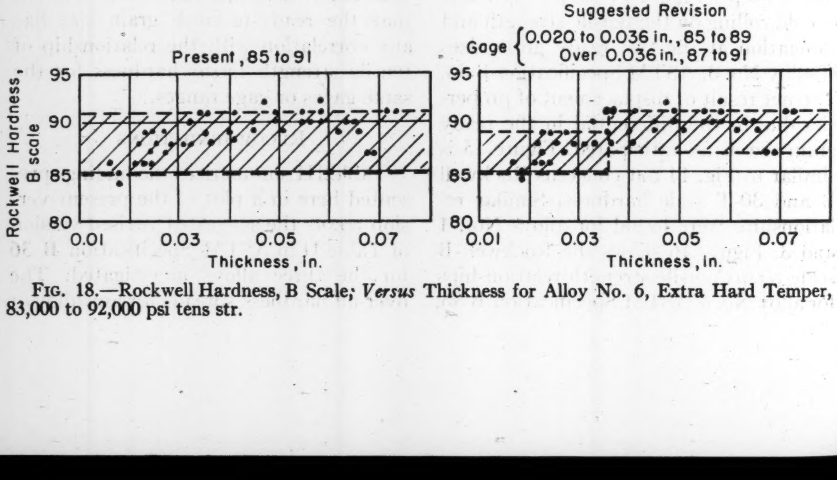


FIG. 18.—Rockwell Hardness, B Scale; *Versus* Thickness for Alloy No. 6, Extra Hard Temper, 83,000 to 92,000 psi tens str.

temper remained unchanged. The 30-T scale values have been adjusted upward by 2 to 3 points as justified by earlier discussions relative to the correct conversion values for the Rockwell B hardness and 30-T superficial hardness scale for the alloy. The hardness spread between the gage ranges at the maximum tensile strength for each rolled temper as shown in Figs. 5 to 10 was used to compile the suggested revision. For example, Fig. 9 shows a maximum tensile strength of 74,000 psi for alloy No. 6, three-quarter hard temper. At this tensile strength, the spread is 3 points on the B scale. Table III shows that this 3-point spread has been maintained for this temper, that is 72 to 79 and 75 to 82 on the B scale. As stated earlier for the 30-T scale, alloy 6, spring and extra spring tempers, only a 2-point spread is noted, not differentiated by gage in this respect. However, the data indicate that a 3-point spread is justified.

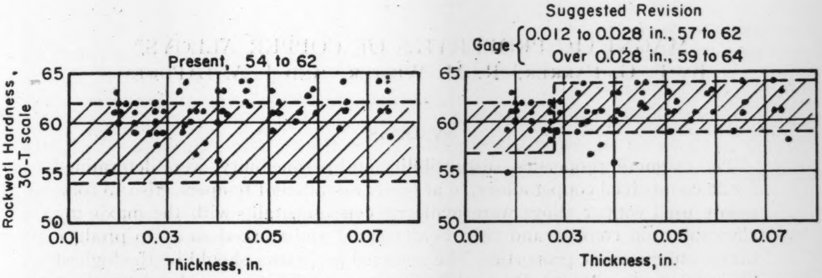


FIG. 19.—Rockwell Superficial Hardness, 30-T Scale, *Versus* Thickness for Alloy No. 1, Hard Temper, 50,000 to 59,000 psi tens str.

In conclusion, Figs. 18 and 19 are shown which compare the present hardness values with the suggested revision as shown in Table III. Figure 18 applies to extra hard temper of alloy No. 6; Fig. 19 applies to hard temper of alloy No. 1. These figures show the displacement range of the mean hardness value and spread in the presently applicable specification and the compensation for the displacement and the gage breakdown in the plot of the suggested revision.

APPENDIX III

MAGNETIC PROPERTIES OF COPPER ALLOYS*

BY F. G. PARKER,[1] R. M. WINTER,[1] AND J. A. LAPORTA[1]

SYNOPSIS

The magnetic properties, susceptibility and permeability, were determined for 26 commercial copper alloys, in at least two different tempers. Also 13 commonly used copper alloys were made up experimentally with the maximum allowable iron content and then heat treated and worked so as to produce maximum magnetic properties. The reported properties should be the highest likely to be encountered. Permeability and susceptibility were also determined for 6 aluminum bronzes.

A study was made of the effect on magnetic properties of the precipitation of iron in copper, alpha brass, and beta brass. A series of 4 iron contents in copper, 3 iron contents in alpha brass, and 2 iron contents in beta brass were made. Susceptibility and remanence were determined. Tests were made on specimens solution treated and aged, and solution treated, worked, and aged. Aging temperatures used were 400 and 600 C with times up to 250 hr.

The results of this investigation will be reported in two sections, the first to provide data on the magnetic properties of commercial copper alloys and the second a presentation of magnetic studies on the effect of precipitation of iron in copper, alpha brass, and beta brass.

Copper alloys are generally considered to be nonmagnetic, but under certain conditions they show definite magnetic properties. These magnetic characteristics are most often associated with the iron content of the alloys and the mode of its occurrence. They are undesirable in alloys to be used in certain equipment or instruments such as compasses or magnetic devices. The magnetic requirements will vary according to the particular use under consideration. There are various specifications with maximum allowable magnetic permeability ranging from 1.02 to 2.0 cgs units. The first part of this work was undertaken in order to determine what commercial alloys would meet these specifications.

TERMINOLOGY AND EQUATIONS

Since all metallurgists may not be generally familiar with magnetic terms and relationships the common terminology is listed here. All definitions are given in the electromagnetic cgs system. If a substance is placed in a magnetic field, the intensity of magnetization in the substance may be slightly smaller, or somewhat larger, than that produced in a vacuum by the same field (Fig. 1). In the first case the substance is called diamagnetic; in the second, paramagnetic. There is also the case of ferromagnetism in which the intensity of magnetization may be very much larger. Ferromagnetic materials will retain a certain amount of the magnetism after the field is removed.

---

* Presented at a meeting of Committee B-5 on Copper and Copper Alloys, Washington, D. C., Sept. 23, 1959.

[1] Research and Development Dept., Chase Brass and Copper Co., Inc., Waterbury, Conn.

Unit Pole.—A unit pole gives rise to a unit field everywhere on the surface of a sphere of unit radius enclosing the pole, and this sphere has an area of 4π. A unit pole separated from a pole of equal strength by a distance of 1 cm will exert a force of 1 dyne.

Field Intensity, H.—Expressed in oersteds. A field of 1 oersted is of such strength that a unit pole placed in it experiences a force of 1 dyne. Magnetic field intensity is like pressure in a water main or the voltage in an electrical circuit, being a measure of the magnetic potential.

Flux, ϕ.—The number of lines of induction emerging from a given pole. The unit of flux is a maxwell. It is analogous to the current in a wire, or the quantity of water flowing in a pipe. In vacuum the number of lines of induction are equivalent to the number of lines of force.

Flux Density, B.—The number of lines of induction per unit area. The unit is the maxwell per sq cm, known as a gauss. Since a unit pole represents 4π line of force surrounding it, it also possesses 4π maxwells of induction.

Intensity of Magnetization, I.—Magnetic moment per unit of volume.

Permeability, u, $= \frac{B}{H}$.—The ratio of the flux density to the field intensity producing it. Since $u = 1$ for a vacuum it also represents the ratio of the flux density of a given material to that of a vacuum produced by a given field intensity. In diamagnetic and paramagnetic materials the ratio of flux density to field intensity is constant. In ferromagnetic materials this is not a linear function until the material becomes saturated.

Volume Magnetic Susceptibility, $K = \frac{I}{H}$.—The ratio of the magnetic moment per unit volume of substance to the applied field intensity.

Specific or Mass Susceptibility, $X = \frac{K}{\rho}$, where ρ = density *g* per cu cm.—The susceptibility per gram of substance.

Remanence.—The residual magnetic moment per unit of volume after the field is removed. It is used to distinguish ferromagnetism from paramagnetism in alloys with small amounts of iron.

The magnetic properties of copper alloys which would be of most interest are the susceptibility (K or X) and the remanence. The most commonly used property is the permeability, u, which is readily calculated from the susceptibility.

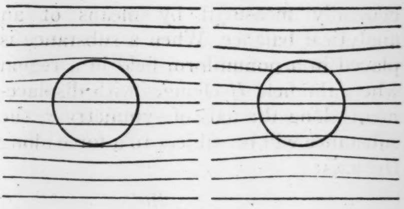

(*a*) (*b*)

FIG. 1.—Permeability of (*a*) Diamagnetic and (*b*) Paramagnetic Bodies.

Diamagnetic bodies are less permeable than a vacuum to magnetic lines of force; paramagnetic bodies are more permeable than a vacuum.

If a substance is placed in a field of strength H, then the magnetic induction is given by B, where:

$$B = H + (4\pi)I \ldots\ldots\ldots\ldots (1)$$

where H = field intensity and I = intensity of magnetization.

$$\text{Permeability } \frac{B}{H} = 1 + 4\pi\frac{I}{H} \ldots\ldots (2)$$

$$\frac{I}{H} = K, \text{ magnetic susceptibility per unit volume} \ldots (3)$$

$$\text{Permeability, } u = 1 + 4\pi K \ldots\ldots (4)$$

$$\frac{K}{d} = X \ldots\ldots\ldots\ldots\ldots (5)$$

$$\text{Permeability, } u = 1 + 4\pi X\rho \ldots\ldots (6)$$

Since the amount of iron present in copper alloys is generally small, the methods most suitable for magnetic measurements on copper alloys are those employed for testing diamagnetic and paramagnetic properties of materials. The apparatus most often employed for susceptibility measurements uses the principle of measuring the force acting upon a known mass of the substance in a nonuniform magnetic field. The force is generally measured by means of an analytical balance. When a substance is placed in a nonuniform field in a region where the field H changes with displacement along the axis of symmetry s, the substance will be subject to a force along the axis:

$$f = mXH \frac{dH}{ds} \ldots\ldots\ldots\ldots (7)$$

where:

f = force in dynes,
X = mass susceptibility,
m = mass of the specimen,
H = field strength, and
$\frac{dH}{ds}$ = the field gradient along the s axis.

The expression for mass susceptibility can then be written:

$$X = \frac{f}{mH \frac{dH}{ds}} \ldots\ldots\ldots\ldots (8)$$

From this equation it is noted that the important feature of the apparatus is to maintain the term $H \frac{dH}{ds}$ constant in the region of the specimen. Equation 8 can be expressed in a more convenient form:

$$X = K \frac{P}{W} \ldots\ldots\ldots\ldots (9)$$

where:

K = constant,
P = pull in grams in the magnetic field, and
W = weight of specimen.

Equation 9 is used in these studies to determine susceptibility from the weight and the pull, as measured by the weight in the field minus the weight in air. K, the constant in the equation, is determined by running some material of known susceptibility and substituting its value in the equation. The advantages of this method are that only a small specimen is required and the specimen can be of irregular shape.

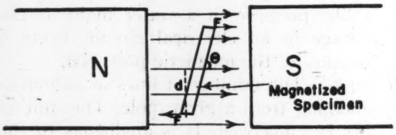


FIG. 2.—Schematic Relation of Forces in a Magnetic Field.

Permeability is then calculated from X by Eq. 6.

Remanence measurements are made by suspending a previously magnetized specimen at right angles to a known uniform field and measuring the torque exerted on the specimen by means of a torsion balance. A magnetized specimen placed at right angles to a known uniform field tries to align itself in the direction of the field. The torque is calculated as shown schematically in Fig. 2.

Since:

$$F = mH \ldots\ldots\ldots\ldots (10)$$

where F is the force in dynes exerted on the ends of the specimen tending to rotate it into the direction of the field, m is the pole strength of the specimen in cgs units, and H is the magnetic field in oersteds, the torque exerted is:

$$T = 2\,F\,d \ldots\ldots\ldots\ldots (11)$$

and since $d = \frac{l}{2} \sin \theta$ where l is the length of the specimen in cm, and θ is the angle of deflection:

$$T = 2\,F\frac{l}{2}\sin\theta \,.........\, (12)$$

$$= m\,l\,H\sin\theta \,.........\, (13)$$

$$= M\,H\sin\theta$$

where $M = ml$ is the magnetic moment of the specimen. This relationship is generally expressed:

$$T = I\,VH\sin\theta \,........\, (14)$$

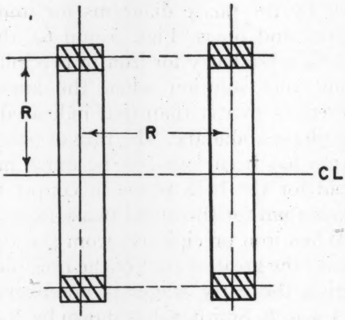


FIG. 3.—Helmholtz Coils for Producing a Uniform Field.

where:

I = the magnetic moment per unit volume in cgs units, and
V = volume of the specimen in cubic centimeters.

If the torque is measured by means of a torsion fiber, the value of I is given by:

$$I = \frac{K\theta'}{l'VH\sin\theta} \,..........\, (15)$$

where:

K = the torsion constant in dyne-sq cm/radian,
θ' = angle of twist in radians, and
l' = the length of the torsion fiber in centimeters.

In measuring θ' the current is reversed and deflections read in each direction and averaged. The reason for reversing is to eliminate deflections due to susceptibility and the shape of the specimen. Equation 15 is used in these studies to determine remanence.

A uniform field over a large space is best produced by means of a set of Helmholtz coils. These consist of two circular coils spaced and dimensioned as shown in Fig. 3, with equal currents flowing in the same sense in the two coils.

MAGNETIC PROPERTIES OF COMMERCIAL COPPER ALLOYS

The object of this part of the investigation was to determine the magnetic susceptibility of various commercial copper alloys, some commonly used copper alloys with maximum allowable iron content, and some aluminum bronzes. The mass susceptibility has been determined for each alloy and the permeability calculated. Various tempers and heat treatments have been included. The magnetic properties, analysis, and history of these alloys are given in Tables I to VII.

The mass susceptibility of copper alloys depends not only on the amount of iron present but also on the form, size, and distribution of the iron. These latter conditions are influenced to a considerable degree by the alloying elements other than iron present and the thermal and mechanical history of the specimen.

Previous Work:

The first extensive investigation of magnetic characteristics of commercial copper alloys was presented by Butts and Rieber in 1949 (**1**).[2] Their paper lists the mass susceptibility and remanent magnetic moment of 10 commercial cast alloys and 10 commercial wrought alloys with iron contents ranging from 0.01 to

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

0.85 per cent. Their data indicate that the magnetic properties of copper alloys are essentially related to the iron content which may be present as an impurity or is intentionally added as an alloying element. The amount of magnetism obtained with a given amount of iron was found to depend on the form in which the iron was present, that is, whether in solid solution, precipitated in a nonmagnetic form, or precipitated in a magnetic form.

For most copper alloys the relationship between magnetic susceptibility and iron content followed a curve shown schematically in Fig. 4. This curve shows three characteristic stages: (1) an initial stage where there is a slow increase in magnetic susceptibility with iron content, (2) a second stage of more rapid rise, and (3) a third stage of very rapid rise.

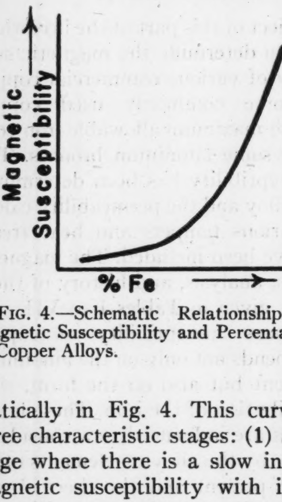


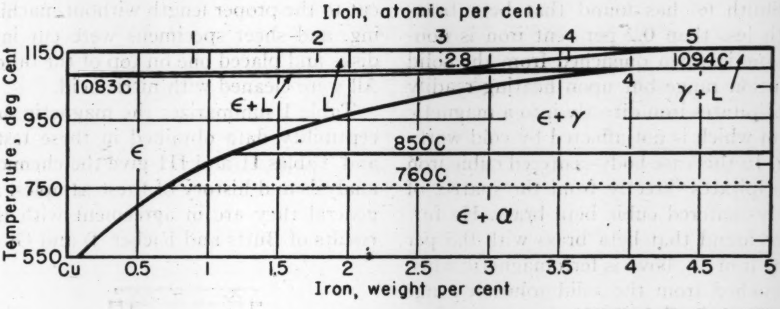
FIG. 4.—Schematic Relationship Between Magnetic Susceptibility and Percentage of Iron in Copper Alloys.

The exact susceptibility value for a given iron content depends on the composition of the alloy and on its previous thermal and mechanical history. In terms of magnetic instruments, Butts and Rieber considered that alloys with magnetic properties below the knee of the curve were acceptable for instruments of high precision, those with magnetic properties at the knee for instruments of lesser precision, while those on the steep part of the curve were unacceptable where magnetic properties might be a source of trouble. In general, if the iron content was kept below 0.01 per cent the magnetic properties of most copper alloys would fall in the first stage of the curve irrespective of alloy content or previous thermal or mechanical history.

It has been established that iron in solid solution in copper alloys does not result in ferromagnetic properties (2–7). However, since the solubility of iron in copper alloys generally decreases very rapidly with decrease in temperature as seen by the phase diagrams for iron in copper and brass (Figs. 5 and 6), there will be a tendency for iron to precipitate from solid solution when the amount present is greater than that indicated by the phase boundary. The rate of precipitation has been found to be much more rapid for the beta phase in copper-zinc alloys than for the alpha phase (6, 8).

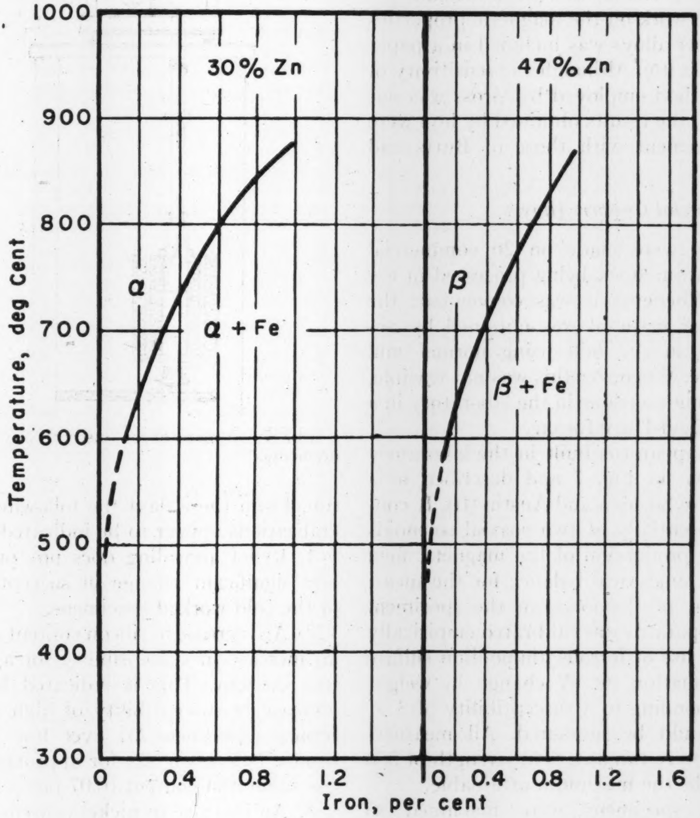
When iron precipitates from the alpha phase, the greatest part of the precipitate during the early stages is nonmagnetic (2, 3, 4, 6, 7). Smith (4) has shown by X-ray analysis that this nonmagnetic iron precipitate possesses a face-centered cubic lattice which matches the lattice of the copper matrix. Cold working (2, 4, 6, 7) or quenching to temperatures appreciably below room temperature (9) will convert the nonmagnetic face-centered cubic structure to the ferromagnetic body-centered cubic structure (4).

From the above it is evident that it is possible to render a magnetic copper alloy nonmagnetic by heating to a temperature sufficiently high to take the iron into solid solution and then quenching so as to retain it in this state. Alternatively, by slow cooling from an elevated temperature or by aging at a lower temperature iron will precipitate, giving rise to magnetism which can be greatly accentuated by cold working.



ASM Metals Handbook, 1948.

FIG. 5.—Solubility of Iron in Copper.



Bauer and Hansen (8).

FIG. 6.—Solubility of Iron in Alpha and Beta Brass.

Smith (6) has found that beta brass with less than 0.2 per cent iron is nonmagnetic when quenched from the solid solution range but upon heating readily precipitates iron directly into a magnetic form which is not affected by cold working. In this case body-centered cubic iron precipitates directly from the matrix of body-centered cubic beta brass. He further found that beta brass with 0.3 per cent iron or above is ferromagnetic when quenched from the solid solution range and becomes less magnetic as more iron precipitates. No definite explanation could be given for this phenomenon.

Some work on the magnetic properties of copper alloys was included in a paper by Gross (10). Although the sensitivity of the method employed by Gross was not as high, the results obtained by him were in agreement with those of Butts and Rieber.

Commercial Copper Alloys:

Tests were made on 26 commercial alloys from stock being processed in the mill. Whenever it was convenient, the annealed material was obtained by annealing in the mill using normal mill practice. Whenever this was not feasible, annealing was done in the laboratory in a small Hevi-Duty furnace.

The apparatus built in the laboratory is shown in Fig. 7 and described in a paper by Sosman and Austin (11). It consists essentially of two coaxial solenoids for the production of the magnetic field and an analytical balance for the measurement of the force on the specimen. The apparatus was calibrated empirically by the use of ferrous ammonium sulfate and equation (9). A change in weight corresponding to a susceptibility of 3 × 10^{-8} could be measured. All measurements were run at a field strength of 536 oersteds, the maximum attainable.

Rod specimens were machined to proper diameters, tube specimens were cut to the proper length without machining, and sheet specimens were cut into disks and placed one on top of the other. All were cleaned with nitric acid.

Table I summarizes the magnetic susceptibility data obtained in these tests, and Tables II and III give the chemical analysis and history of these samples. In general they are in agreement with the results of Butts and Rieber (1) and Gross (10). From these data the following generalizations appear to be indicated:

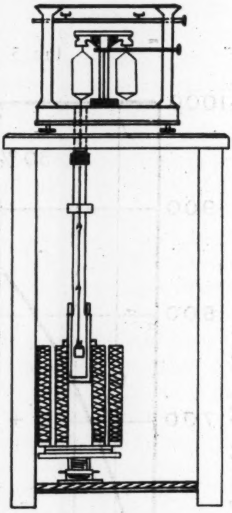


FIG. 7.—Apparatus for Susceptibility Measurements.

1. Relief annealing does not produce any significant change in susceptibility of the cold worked specimens.
2. An increase in silicon content causes an increase in susceptibility for a given iron content. This is indicated by the increase in susceptibility of high silicon bronze (specimen 23) over low silicon bronze (specimen 22) for approximately the same iron content (0.07 per cent).
3. An increase in nickel content tends to nullify the effects of iron in producing

TABLE I.—MASS SUSCEPTIBILITY OF COPPER ALLOYS.

Specimen Number	Alloy	Temper	Rockwell Hardness, B Scale	Iron, actual per cent	Iron, max per cent (ASTM Spec.)	Mass Susceptibility, X, cgs units	Permeability, μ, cgs units
	Electrolytic Tough Pitch Copper						—
1....	E.T.P..................	hard	48	0.0035	...	0.66×10^{-6}	1.000074
	Brasses						
2....	Commercial bronze......	hard	68	<0.005	0.05	−0.14	0.999985
2A...	Commercial bronze......	anneal	17	<0.005	0.05	−0.16	0.999982
3....	Low brass..............	hard	82	0.0036	0.05	−0.15	0.999984
3A...	Low brass..............	anneal	22	0.0036	0.05	−0.15	0.999984
4....	Cartridge brass........	hard	82	0.0043	0.05	−0.24	0.999974
4A...	Cartridge brass........	anneal	29	0.0079	0.05	−0.15	0.999984
5....	Yellow brass...........	extruded	16	0.012	0.05	−0.08	0.999991
6....	Muntz metal............	hard	85	0.0048	0.07	−0.17	0.999982
6A...	Muntz metal............	anneal	30	0.0048	0.07	−0.17	0.999982
6B...	Muntz metal............	extruded	42	0.007	0.07	−0.21	0.999978
	Lead-Bearing Brasses						
7....	Leaded commercial bronze............	half hard	63	0.012	0.10	−0.17	0.999981
8....	Free cutting brass.......	half hard	80	0.11	0.35	5.66	1.000604
8A...	Free cutting brass.......	light anneal	25	0.11	0.35	3.41	1.000364
8B...	Free cutting brass.......	half hard	56	0.078	0.35	2.25	1.000240
8C...	Free cutting brass.......	R. A.[a]	62	0.078	0.35	2.23	1.000238
8D...	Free cutting brass.......	light anneal	24	0.078	0.35	0.73	1.000078
8E...	Free cutting brass.......	half hard	76	0.053	0.35	0.33	1.000035
8F...	Free cutting brass.......	half hard	74	0.34	0.35	275.5	1.029400
8G...	Free cutting brass.......	light anneal	34	0.34	0.35	94.85	1.010120
9....	Forging brass..........	extruded	50	0.034	0.30	0.88	1.000093
10....	Architectural bronze......	extruded	58	0.075	...	19.06	1.002029
	Special Brasses						
11....	Naval brass............	hard	85	0.022	0.10	0.64	1.000068
11A...	Naval brass............	R.A.[a]	82	0.022	0.10	0.33	1.000035
11B...	Naval brass............	light anneal	49	0.022	0.10	0.44	1.000047
12....	Leaded naval brass......	hard	85	0.040	0.10	2.89	1.000306
12A....	Leaded naval brass......	R.A.[a]	84	0.040	0.10	3.12	1.000330
12B....	Leaded naval brass......	light anneal	50	0.040	0.10	8.26	1.000875
13....	Manganese bronze.......	extruded	68	1.06	2.00	380.0	1.039910
14....	Inhibited admiralty......	hard	89	0.012	0.06	−0.25	0.999973
14A....	Inhibited admiralty......	anneal	29	0.015	0.06	−0.24	0.999974
15....	Aluminum brass.........	anneal	43	0.015	0.07	−0.34	0.999964

TABLE I.—*Concluded.*

Specimen Number	Alloy	Temper	Rockwell Hardness, B Scale	Iron, actual per cent	Iron, max per cent (ASTM Spec.)	Mass Susceptibility, X, cgs units	Permeability, μ, cgs units
	TIN BRONZES, PHOSPHOR BRONZES						
16....	Phosphor bronze, 5 per cent	cast	52	0.013	0.10	−0.15	0.999983
16A...	Phosphor bronze, 5 per cent	half hard	73	0.003	0.10	−0.16	0.999982
16B...	Phosphor bronze, 5 per cent	R.A.[a]	73	0.003	0.10	−0.10	0.999989
16C...	Phosphor bronze, 5 per cent	anneal	31	0.003	0.10	−0.12	0.999987
17....	Free cutting phosphor bronze	half hard	62	0.024	0.10	0.08	1.000009
17A...	Free cutting phosphor bronze	hard	82	0.054	0.10	11.62	1.001296
17B...	Free cutting phosphor bronze	anneal	27	0.054	0.10	22.90	1.003235
	COPPER-NICKEL ALLOYS						
18....	Nickel silver, 12 per cent	hard	87	0.033	...	0.47	1.000051
18A...	Nickel silver, 12 per cent	soft	45	0.033	...	0.66	1.000072
19....	Nickel silver, 18 per cent	hard	90	0.066	...	0.57	1.000063
19A...	Nickel silver, 18 per cent	soft	36	0.066	...	0.56	1.000062
20....	Cupro-nickel, 10 per cent	hard	69	1.35	2.00	105.5	1.011849
20A...	Cupro-nickel, 10 per cent	anneal	40	1.35	2.00	3525	1.395900
20B...	Cupro-nickel, 10 per cent	hard	82	1.34	2.00	1965	1.134400
20C...	Cupro-nickel, 10 per cent	anneal	27	1.34	2.00	189.7	1.021219
21....	Cupro-nickel, 30 per cent	hard	90	0.63	0.70	5.19	1.000583
21A...	Cupro-nickel, 30 per cent	anneal	39	0.66	0.70	6.72	1.000754
	ENGINEERING ALLOYS						
22....	Low silicon bronze	hard	86	0.072	0.20	0.16	1.000018
22A...	Low silicon bronze	R.A.[a]	84	0.072	0.20	0.18	1.000020
22B...	Low silicon bronze	anneal	20	0.072	0.20	0.00	1.000000
23....	High silicon bronze	hard	94	0.067	0.20	24.79	1.002658
23A...	High silicon bronze	R.A.[a]	101	0.067	0.20	24.95	1.002675
23B...	High silicon bronze	anneal	43	0.067	0.20	−0.08	0.999991
24....	Tellurium copper	hard	48	<0.002	...	−0.08	0.999991
24A...	Tellurium copper	hard	45	<0.001	...	−0.11	0.999988
25....	Telnic bronze	anneal	36	0.013	...	−0.17	0.999981
25A...	Telnic bronze	age harden	88	0.013	...	0.00	1.000000
25B...	Telnic bronze	C.W. + age[b]	79	0.009	...	−0.17	0.999981
26....	Phosnic bronze	anneal	38	0.005	0.10	−0.15	0.999983
26A...	Phosnic bronze	half hard	80	<0.01	0.10	−0.15	0.999983
26B...	Phosnic bronze	age hard	74	<0.005	0.10	−0.07	0.999992
26C...	Phosnic bronze	age + C.W.[c]	86	<0.005	0.10	−0.09	0.999990

[a] R.A. = Relief anneal.

[b] Draw 24 per cent plus age 2½ hr at 850 F.

[c] Age 2½ hr at 850 F plus draw 23 per cent.

abnormally high susceptibility. This is indicated by the relatively low susceptibility of 30 per cent cupro-nickel (specimens 21 and 21A) with approximately 0.6 per cent iron.

4. In $\alpha + \beta$ alloys the susceptibility increases with increase in iron content, giving a very rapid increase beyond a certain iron content.

In these tests no attempt was made to vary the iron content of the various alloys but rather only to test typical alloys as they were being processed in the mill. With one exception (free-cutting brass) the iron contents considered were below the maximum listed in the chemical specifications. Furthermore, no special thermal or mechanical treatments were considered other than those which might occur in mill practice. Consequently the data reported for a given alloy are for the particular iron content and thermal and mechanical history listed. Other iron contents or treatments could produce quite drastic changes in the susceptibility values reported for a given alloy.

Sufficient data to indicate how the susceptibility varies with iron content were obtained only for free-cutting brass. For this alloy specimens were available with iron contents ranging from 0.053 to 0.34 per cent. The susceptibility *versus* iron content in this range is plotted in Fig. 8 for the half-hard and the annealed tempers. Also plotted are the data by Butts and Rieber on the same alloy. The curves show the characteristic form associated with such plots; that is, a slow rise, followed by a more rapid rise, and finally a very steep rise in magnetic susceptibility *versus* iron content. At low iron contents, that is, less than 0.10 per cent iron, the agreement between the present data and that of Butts and Rieber is very good. At higher iron contents, the susceptibility values obtained by Butts and Rieber are somewhat lower than those obtained in the present tests. This is not surprising in view of the extreme sensitivity of the magnetic properties of copper alloys to thermal and mechanical history.

Copper Alloys with Maximum Allowable Iron:

Since it was recognized that the maximum permeability likely to be found in copper alloys was dependent on iron content and previous thermal and mechanical treatment, it was decided to prepare 13 alloys commonly used but with an iron content at or near the maximum allowed by analysis specification. All these alloys were cast, swaged, and annealed in the laboratory.

Susceptibility measurements were made as previously described at 536 oersteds for various treatments of these alloys and reported in Table IV. Tables V and VI give the chemical analysis and history of these specimens. Determinations were made on specimens as finished with approximately a 50 per cent reduction as shown in Table VI, as annealed 800 C and water quenched, the same and then aged for 1 hr at 500 and water quenched, the same and then cold worked 50 per cent, and also as air cooled after aging. These treatments are given in Table IV with corresponding susceptibilities. The maximum value for any one alloy is in italics. In the last column is listed the maximum permeability calculated from the maximum susceptibility. This represents the maximum value likely to be encountered in any of these commercial alloys.

Aluminum Bronzes:

Six specimens of commercial aluminum bronze were obtained in the extruded, drawn, annealed, and straightened condition. Analysis, susceptibility, permeability, and mechanical properties are

TABLE II.—COMPOSITION OF COPPER ALLOYS USED FOR MAGNETIC MEASUREMENTS.

Specimen Number	Alloy	Iron, per cent	Copper, per cent	Lead, per cent	Nickel, per cent	Tin, per cent	Phosphorus, per cent	Aluminum, per cent	Silicon, per cent	Manganese, per cent	Antimony per cent	Tellurium, per cent	Zinc, per cent
	ELECTROLYTIC TOUGH PITCH COPPER												
1	E.T.P.	0.0035	99.9+	<0.001	<0.001	<0.001	...	<0.001	<0.001	<0.001	...	...	<0.01
	BRASSES												
2	Commercial bronze	<0.005	89.9	<0.005	0.02	<0.005	...	...	...	...	...	...	10.08
2A	Commercial bronze	<0.005	89.9	<0.005	0.02	<0.005	...	...	...	...	...	...	10.08
3	Low brass	0.0036	80.15	<0.01	<0.002	...	...	<0.001	...	<0.001	...	...	19.85
3A	Low brass	0.0036	80.15	<0.01	<0.002	...	...	<0.001	...	<0.001	...	...	19.85
4	Cartridge brass	0.0079	69.33	<0.01	0.015	...	...	<0.001	...	<0.001	...	...	30.65
4A	Cartridge brass	0.0079	69.33	<0.01	0.015	...	...	<0.001	...	<0.001	...	...	30.65
5	Yellow brass	0.012	66.52	0.007	0.02	0.002	...	...	...	...	...	...	33.44
6	Muntz metal	0.0048	62.26	<0.01	0.017	...	...	<0.001	...	<0.001	...	...	37.72
6A	Muntz metal	0.0048	62.26	<0.01	0.017	...	...	<0.001	...	<0.001	...	...	37.72
6B	Muntz metal	0.007	62.21	0.008	0.013	0.002	...	...	...	...	...	...	37.76
	LEAD-BEARING BRASSES												
7	Leaded commercial bronze	0.012	88.99	2.05	0.015	0.012	...	...	...	...	...	...	8.92
8	Free cutting brass	0.11	60.37	3.33	0.068	0.066	...	...	...	...	...	...	36.06
8A	Free cutting brass	0.11	60.37	3.33	0.068	0.066	...	...	...	...	...	...	36.06
8B	Free cutting brass	0.078	61.93	3.09	0.076	0.070	...	...	...	...	...	...	34.76
8C	Free cutting brass	0.078	61.93	3.09	0.076	0.070	...	...	...	...	...	...	34.76
8D	Free cutting brass	0.078	61.93	3.09	0.076	0.070	...	...	...	...	...	...	34.76
8E	Free cutting brass	0.053	61.56	3.19	0.019	0.048	...	<0.005	<0.005	<0.005	...	...	35.13
8F	Free cutting brass	0.34	61.97	3.26	0.11	0.090	...	<0.001	<0.01	<0.005	<0.01	...	34.23
8G	Free cutting brass	0.34	61.97	3.26	0.11	0.090	...	<0.001	<0.01	<0.005	<0.01	...	34.23
9	Forging brass	0.034	59.78	1.91	0.037	0.044	...	...	...	...	...	...	38.19
10	Architectural bronze	0.075	58.66	2.94	0.028	0.087	...	<0.005	<0.005	<0.005	...	...	38.21
11	Naval brass	0.022	60.16	0.054	0.016	0.66	...	...	...	...	0.01	...	39.08
11A	Naval brass	0.022	60.16	0.054	0.016	0.66	...	...	...	...	0.01	...	39.08
11B	Naval brass	0.022	60.16	0.054	0.016	0.66	...	...	...	...	0.01	...	39.08
12	Leaded naval brass	0.040	60.18	1.89	0.027	0.66	...	0.049	...	...	<0.01	...	37.15
12A	Leaded naval brass	0.040	60.18	1.89	0.027	0.66	...	0.049	...	...	<0.01	...	37.15
12B	Leaded naval brass	0.040	60.18	1.89	0.027	0.66	...	0.049	...	...	<0.01	...	37.15
13	Manganese bronze	1.06	58.27	0.05	0.013	0.94	...	...	...	0.22	...	...	39.45
14	Inhibited admiralty	0.012	71.38	0.034	0.012	1.02	...	<0.001	...	...	0.031	...	27.51
14A	Inhibited admiralty	0.015	71.44	0.032	0.016	1.02	...	<0.001	...	...	0.03	...	27.45
15	Aluminum brass	0.015	76.65	0.016	0.019	0.089	...	1.88	<0.01	<0.01	0.008	...	21.32

					TIN BRONZES, PHOSPHOR BRONZES								
16	Phosphor bronze, 5 per cent	0.013	94.75	<0.01	<0.01	5.01	0.23	...	...	...	...	...	<0.01
16A	Phosphor bronze, 5 per cent	0.003	93.83	<0.01	<0.002	5.94	0.23	<0.001	...	<0.001	...	...	<0.01
16B	Phosphor bronze, 5 per cent	0.003	93.83	<0.01	<0.002	5.94	0.23	...	...	...	...	...	...
16C	Phosphor bronze, 5 per cent	0.003	93.83	<0.01	<0.002	5.94	0.23	...	...	...	...	...	...
17	Free cutting phosphor bronze	0.024	87.84	3.76	0.048	4.19	0.048	...	...	...	0.061	...	4.03
17A	Free cutting phosphor bronze	0.054	88.40	3.97	0.054	3.97	0.110	...	...	...	0.050	...	3.39
17B	Free cutting phosphor bronze	0.054	88.40	3.97	0.054	3.97	0.110	...	...	...	0.050	...	3.39
					COPPER-NICKEL ALLOYS								
18	Nickel silver, 12 per cent	0.033	62.28	0.94	13.18	<0.01	...	<0.001	<0.001	0.19	<0.01	...	23.38
18A	Nickel silver, 12 per cent	0.033	62.28	0.94	13.18	<0.01	...	<0.001	<0.001	0.19	<0.01	...	...
19	Nickel silver, 18 per cent	0.066	62.07	0.94	17.42	<0.01	...	<0.001	<0.001	0.27	<0.01	...	19.23
19A	Nickel silver, 18 per cent	0.066	62.07	0.94	17.42	<0.01	...	<0.001	<0.001	0.27	<0.01	...	...
20	Cupro-nickel, 10 per cent	1.35	88.40	<0.01	9.80	...	...	...	...	0.27	...	...	0.18
20A	Cupro-nickel, 10 per cent	1.35	88.40	<0.01	9.80	...	...	...	...	0.27	...	...	0.18
20B	Cupro-nickel, 10 per cent	1.34	88.30	<0.01	10.00	...	...	...	...	0.16	...	...	0.20
20C	Cupro-nickel, 10 per cent	1.34	88.33	<0.01	9.89	...	...	...	...	0.34	...	...	0.098
21	Cupro-nickel, 30 per cent	0.63	68.66	<0.01	29.84	...	...	...	...	0.70	...	...	0.17
21A	Cupro-nickel, 30 per cent	0.66	68.69	<0.01	29.74	...	...	...	...	0.70	...	...	0.21
					ENGINEERING ALLOYS								
22	Low silicon bronze	0.072	96.79	0.012	0.02	0.14	...	<0.005	1.32	0.035	...	...	1.61
22A	Low silicon bronze	0.072	96.79	0.012	0.02	0.14	...	<0.005	1.32	0.035	...	...	...
22B	Low silicon bronze	0.072	96.79	0.012	0.02	0.14	...	<0.005	1.32	0.035	...	...	...
23	High silicon bronze	0.067	96.55	<0.01	0.038	0.022	...	...	3.11	...	...	...	0.26
23A	High silicon bronze	0.067	96.55	<0.01	0.038	0.022	...	...	3.11	...	...	...	0.26
23B	High silicon bronze	0.067	96.55	<0.01	0.038	0.022	...	...	3.11	...	...	...	0.26
24	Tellurium copper	<0.002	99.36	0.02	0.05	0.002	0.046	<0.002	...	...	...	0.43	0.09
24A	Tellurium copper	<0.001	99.61	negative	negative	...	0.004	...	...	...	...	0.39	negative
25	Telnic bronze	0.013	98.04	<0.01	1.21	<0.005	0.24	...	...	...	...	0.50	<0.01
25A	Telnic bronze	0.013	98.04	<0.01	1.21	<0.005	0.24	...	...	...	...	0.50	...
25B	Telnic bronze	0.009	97.94	0.024	1.00	0.017	0.25	<0.005	<0.005	<0.005	...	0.52	0.25
26	Phosnic bronze	0.005	98.59	0.013	1.04	0.08	0.25	...	...	...	...	0.023	<0.01
26A	Phosnic bronze	<0.01	98.31	0.017	1.04	0.068	0.26	<0.001	<0.002	<0.002	...	<0.05	0.30
26B	Phosnic bronze	<0.005	98.65	<0.01	1.10	<0.01	0.21	<0.001	<0.002	<0.002	...	<0.05	0.036
26C	Phosnic bronze	0.005	98.65	<0.01	1.10	<0.01	0.21	<0.001	<0.002	<0.002	...	<0.05	0.036

TABLE III.—HISTORY OF COPPER ALLOYS USED FOR MAGNETIC MEASUREMENTS.

Specimen Number	Alloy	Form	Size, in.	Temper
ELECTROLYTIC TOUGH PITCH COPPER				
1	E.T.P.	rod	1.00	drawn 36 per cent
BRASSES				
2	Commercial bronze	rod	0.625	drawn 30 per cent
2A	Commercial bronze	rod	0.625	drawn 30 per cent, mill anneal 2 hr at 1000 F
3	Low brass	rod	0.625	drawn 43 per cent
3A	Low brass	rod	0.625	drawn 43 per cent, mill anneal 2 hr at 1000 F
4	Cartridge brass	rod	0.562	drawn 33 per cent
4A	Cartridge brass	rod	0.562	drawn 33 per cent, mill anneal 2 hr at 1000 F
5	Yellow brass	rod	0.750	extruded
6	Muntz metal	rod	0.500	drawn 33 per cent
6A	Muntz metal	rod	0.500	drawn 33 per cent, mill anneal 2 hr at 1000 F
6B	Muntz metal	rod	0.625	extruded
LEAD-BEARING BRASSES				
7	Leaded commercial bronze	rod	1.250	drawn 30 per cent
8	Free cutting brass	rod	0.500	drawn 23 per cent
8A	Free cutting brass	rod	0.500	Drawn 23 per cent, mill anneal 75 min at 1200 F
8B	Free cutting brass	rod	1.062	drawn 11 per cent
8C	Free cutting brass	rod	1.062	drawn 11 per cent, laboratory relief anneal 30 min at 500 F
8D	Free cutting brass	rod	1.062	drawn 11 per cent, laboratory anneal 1 hr at 1200 F
8E	Free cutting brass	rod	0.609	drawn 19 per cent
8F	Free cutting brass	rod	0.687	drawn 16 per cent
8G	Free cutting brass	rod	0.687	drawn 16 per cent, mill anneal 75 min at 1200 F
9	Forging brass	rod	0.500	extruded
10	Architectural bronze	shape		extruded
SPECIAL BRASSES				
11	Naval brass	rod	0.750	drawn 14 per cent
11A	Naval brass	rod	0.750	drawn 14 per cent, laboratory relief anneal 30 min at 500 F
11B	Naval brass	rod	0.750	drawn 14 per cent, laboratory anneal 1 hr at 1100 F
12	Leaded naval brass	rod	1.00	drawn 19 per cent
12A	Leaded naval brass	rod	1.00	drawn 19 per cent, laboratory relief anneal 30 min at 500 F
12B	Leaded naval brass	rod	1.00	drawn 19 per cent, laboratory anneal 1 hr at 1100 F
13	Manganese bronze	rod	1.00	extruded
14	Inhibited admiralty	tube	1 × 0.049	drawn 43 per cent
14A	Inhibited admiralty	tube	1 × 0.049	drawn 43 per cent, mill anneal 15 min at 1000 F
15	Aluminum brass	tube	½ × 0.032	drawn 27 per cent, mill anneal 30 min at 1000 F

TABLE III.—*Concluded.*

Specimen Number	Alloy	Form	Size, in.	Temper
Tin Bronzes, Phosphor Bronzes				
16	Phosphor bronze, 5%	rod	0.750	continuous cast
16A	Phosphor bronze, 5%	rod	0.562	drawn 16 per cent
16B	Phosphor bronze, 5%	rod	0.562	drawn 16 per cent, laboratory relief anneal 30 min at 500 F
16C	Phosphor bronze, 5%	rod	0.562	drawn 16 per cent, laboratory anneal 2 hr at 1100 F
17	Free cutting phosphor bronze	rod	1.00	drawn 28 per cent
17A	Free cutting phosphor bronze	rod	1.00	drawn 28 per cent
17B	Free cutting phosphor bronze	rod	1.00	drawn 28 per cent, laboratory anneal 2 hr at 1350 F
Copper-Nickel Alloys				
18	Nickel silver, 12%	rod	0.500	hard
18A	Nickel silver, 12%	rod	0.500	hard, laboratory anneal 1 hr at 1400 F
19	Nickel silver, 18%	rod	0.500	hard
19A	Nickel silver, 18%	rod	0.500	hard, laboratory anneal 1 hr at 1400 F
20	Cupro-nickel, 10%	rod	0.625	drawn 32 per cent
20A	Cupro-nickel, 10%	rod	0.625	drawn 32 per cent, mill anneal 2 hr at 1400 F
20B	Cupro-nickel, 10%	tube	0.875 × 0.049	drawn 20 per cent
20C	Cupro-nickel, 10%	tube	0.875 × 0.049	drawn 20 per cent, mill anneal 130 min at 1300 F
21	Cupro-nickel, 30%	tube	1 × 0.049	drawn 29 per cent
21A	Cupro-nickel, 30%	tube	1 × 0.049	drawn 29 per cent, mill anneal 30 min at 1600 F
Engineering Alloys				
22	Low silicon bronze	rod	0.620	drawn 56 per cent
22A	Low silicon bronze	rod	0.620	drawn 56 per cent, laboratory relief anneal 1 hr at 600 F
22B	Low silicon bronze	rod	0.620	drawn 56 per cent, laboratory anneal 1 hr at 1200 F
23	High silicon bronze	sheet	0.090	cold rolled 38 per cent
23A	High silicon bronze	sheet	0.090	cold rolled 38 per cent, laboratory relief anneal 1 hr at 600 F
23B	High silicon bronze	sheet	0.090	cold rolled 38 per cent, laboratory anneal 1 hr at 1200 F
24	Tellurium copper	rod	0.750	drawn 35 per cent
24A	Tellurium copper	rod	0.875	drawn 40 per cent
25	Telnic bronze	rod	0.500	solution anneal at 1450 F, draw 39 per cent, age 1½ hr at 780 F, mill anneal 1 hr at 1100 F
25A	Telnic bronze	rod	0.500	solution anneal at 1450 F, age 1½ hr at 780 F
25B	Telnic bronze	rod	0.875	solution anneal at 1450 F, draw 24 per cent, age 2½ hr at 850 F
26	Phosnic bronze	rod	0.630	drawn 29 per cent, mill anneal 2 hr at 1200 F
26A	Phosnic bronze	rod	0.625	drawn 30 per cent
26B	Phosnic bronze	rod	0.625	drawn 30 per cent, solution anneal 2½ hr at 1500 F, age 2½ hr at 850 F
26C	Phosnic bronze	rod	0.625	drawn 30 per cent, solution anneal 2½ hr at 1500 F, age 2½ hr at 850 F, plus draw 23 per cent

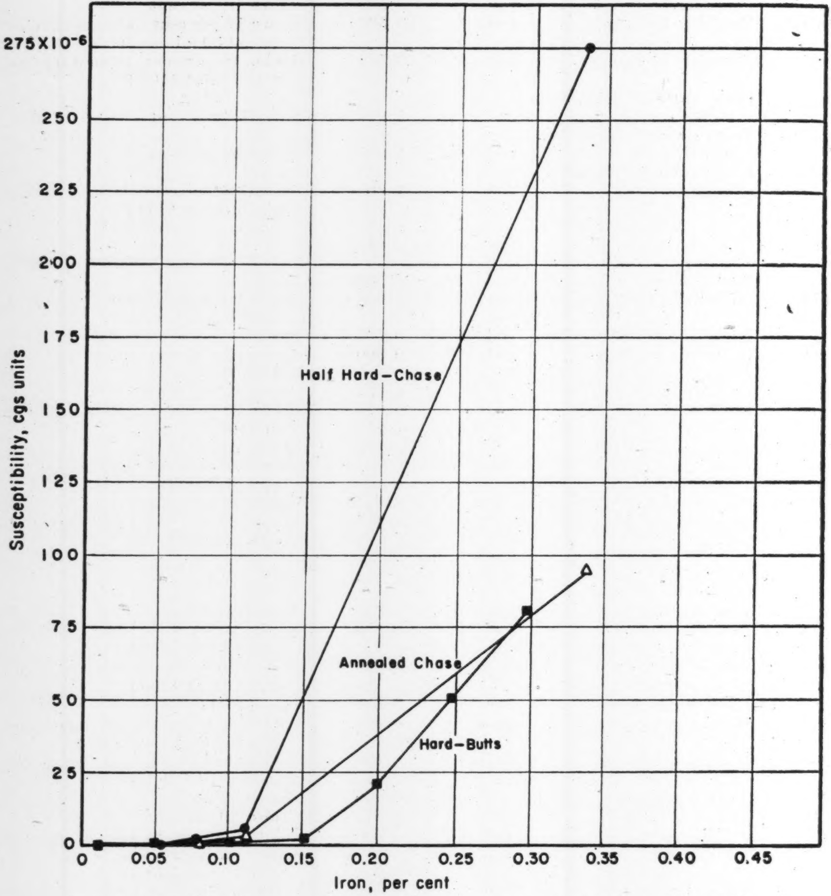


FIG. 8.—Susceptibility as a Function of Iron Content in Free-Cutting Brass Rod.

TABLE IV.—SUSCEPTIBILITY, CGS UNITS.

Alloy,	Iron, per cent	Original 50 per cent Reduction	800 C for 1 hr, WQ	800 C 1 hr, WQ; 500 C. 1 hr, WQ	800 C 1 hr, WQ; 500 C 1 hr, WQ; Cold Worked 50 per cent	500 C 1 hr, Air Cooled	Maximum Permeability, cgs units
Commercial bronze	0.039	0.00×10^{-6}	0.00×10^{-6}	0.00×10^{-6}	0.00×10^{-6}	0.00×10^{-6}	1.000000
Cartridge brass	0.05	0.00	0.00	0.00	0.00	*0.945*	1.000101
Muntz metal	0.07	0.00	1.04	14.5	*15.8*	13.3	1.001663
Leaded commercial bronze	0.112	*59.3*[a]	0.00	...	0.00	6.45	1.006560
Free cutting brass	0.35	11.6	130.5	48.1	*474*	252	1.050600
Architectural bronze	0.15	80.5	60.8	43.1	*139*	57.2	1.014800
Naval brass	0.087	*59.1*	27.4	18.9	37.2	26.0	1.006250
Phosphor bronze 5 per cent	0.085	0.00	*6.6*	1.5	5.7	0.00	1.000737
Phosphor bronze 8 per cent	0.07	0.285	*5.83*	1.42	0.00	1.96	1.000652
Nickel silver 65-18	0.25	1.07	0.97	*1.91*	0.00	0.665	1.000208
Telnic bronze	0.17	0.152	*1.36*	0.00	0.00	0.00	1.000153
Aluminum-silicon bronze	0.38	0.00	1.46	*76*	...	0.00	1.007990
High silicon bronze	0.29	5.95	0.00	*91*	22.8	40.5	1.010000

[a] Maximum value in italics.

TABLE V.—COMPOSITION OF COPPER ALLOYS USED FOR MAGNETIC MEASUREMENT.

Alloy	Copper, per cent	Zinc, per cent	Iron, per cent	Lead, per cent	Nickel, per cent	Tin, per cent	Phosphorus, per cent	Aluminum, per cent	Silicon, per cent	Tellurium, per cent
Commercial bronze	90.24	9.72	0.039	<0.01	...	...	...	...	...	...
Cartridge brass	69.81	29.92	0.05	0.022	<0.01	<0.01	...	...	...	...
Muntz metal	59.94	39.87	0.07	0.012	<0.01	<0.01	...	...	...	...
Leaded commercial bronze	89.08	8.81	0.112	2.09	...	...	...	...	...	...
Free cutting brass	61.91	34.71	0.35	3.03	<0.01	<0.01	...	...	...	...
Architectural bronze	58.68	38.32	0.15	3.00	<0.01	<0.01	...	...	...	...
Naval brass	61.58	37.58	0.087	<0.01	<0.01	0.74	...	...	...	...
Phosphor bronze 5 per cent	94.60	...	0.085	<0.01	...	5.06	0.25	...	...	...
Phosphor bronze 8 per cent	92.27	...	0.07	<0.005	<0.01	7.42	0.24	<0.01	<0.005	...
Nickel silver 65-18	67.47	14.84	0.25	0.015	17.42	<0.01	...	<0.005	<0.005	...
Telnic bronze	98.53	...	0.17	<0.005	0.56	...	0.24	...	...	0.50
Aluminum, silicon bronze	91.01	<0.02	0.38	<0.01	<0.01	<0.02	...	7.06	1.55	...
High silicon bronze	96.05	1.36	0.29	0.019	0.01	0.01	...	...	2.26	...

TABLE VI.—HISTORY OF COPPER ALLOYS USED FOR MAGNETIC MEASUREMENTS.

Alloy	Form	Size, in.	Temper					Rockwell Hardness, B Scale
			Cold Worked, per cent	Annealed WQ	Cold Worked, per cent	Annealed WQ	Cold Worked, per cent	
Commercial bronze	rod	0.250	36	1475 F 1 hr	58	1475 F ½ hr	41 ...	51
Cartridge brass	rod	0.438	53	1450 F 1 hr	51	...	...	81
Muntz metal	rod	0.438	27	1450 F 1 hr	66	...	...	90
Leaded commercial bronze	rod	0.250	36	1475 F ½ hr	58	1475 F ½ hr	41	41
Free cutting brass	rod	0.438	45	1450 F 1 hr	51	...	...	77
Architectural bronze	rod	0.438	38	1450 F 1 hr	51	...	...	84
Naval brass	rod	0.438	41	1450 F 1 hr	51	...	...	88
Phosphor bronze 5 per cent	rod	0.250	36	1475 F 1 hr	58	1475 F ½ hr	41	79
Phosphor bronze 8 per cent	rod	0.438	32	1450 F 1 hr	56	...	...	98
Nickel silver 65-18	rod	0.438	38	1450 F 1 hr	51	...	...	83
Telnic bronze	rod	0.438	39	1450 F 1 hr	51	...	...	60
Aluminum silicon bronze	rod	0.452	43	1450 F 1 hr	41	...	...	99
High silicon bronze	rod	0.250	36	1475 F ½ hr	58	1475 F ½ hr	41	72

reported in Table VII. It is noted that magnetic properties are much higher than for brass alloys. Because of their ferromagnetic properties, their susceptibility and permeability vary with the field strength, and values given are maximum determined at saturation in a field of 2500 oersteds.

TABLE VII.—CHEMICAL, MECHANICAL, AND MAGNETIC PROPERTIES OF COMMERCIAL ALUMINUM BRONZES.

Properties	Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Specimen 6
Magnetic Properties:						
Susceptibility,[a] cgs	1030×10^{-6}	2040×10^{-6}	2110×10^{-6}	1690×10^{-6}	1290×10^{-6}	758×10^{-6}
Permeability, cgs	1.103154	1.193774	1.237214	1.156620	1.114487	1.071805
Chemical Composition:						
Copper, per cent	91.06	87.06	85.62	84.94	81.56	81.44
Aluminum, per cent	6.17	9.56	10.55	11.07	13.65	10.10
Iron, per cent	2.27	3.06	3.69	3.68	4.55	2.77
Nickel, per cent	0.22	0.22	0.12	0.23	0.14	4.77
Manganese, per cent	...	...	...	...	...	0.82
Others, per cent	0.28	0.08	0.02	0.08	0.10	0.10
Mechanical Properties:						
Tensile strength, psi	87 200	96 800	126 500	107 500	106 500	113 000
Yield strength, psi	59 800	56 700	52 200	35 250	65 500	72 200
Elongation, per cent in 2 in.	35.5	33.5	10.0	12.5	0.5	10.5
Contraction of area	66.5	53.0	14.0	13.5	0.0	14.8
Hardness, BHN	163	163	192	196	340	212

[a] Magnetic susceptibility was determined at a field strength of 2500 oersteds.

MAGNETIC STUDIES ON THE PRECIPITATION OF IRON IN COPPER, ALPHA BRASS, AND BETA BRASS

Because of the irregularities found in magnetic properties of commercial alloys a project was undertaken for the systematic study of the effect of iron in copper, alpha brass, and beta brass. The alpha plus beta brasses were purposely avoided so as not to have the added uncertainty of variable amounts of alpha and beta.

Materials, Apparatus, and Methods:

Susceptibility and remanence were determined on specimens given various solution and aging heat treatments. To be certain that specimens were saturated, especially the ferromagnetic higher iron contents, a new higher strength magnet was used for this work. Field strengths up to 15,000 oersteds were available with a 1-in. gap. A constant gradient field was obtained by the use of pole pieces shaped as shown in Fig. 9. A change in weight corresponding to a susceptibility of 3×10^{-8} could be measured.

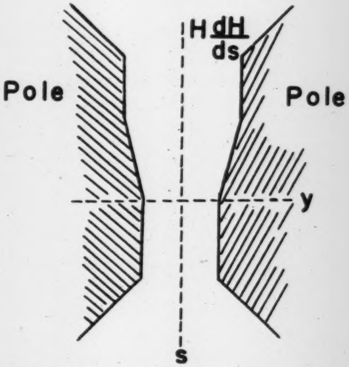


FIG. 9.—Shape of Pole Pieces for Constant Gradient Field.

The curve in Fig. 10 of a 1 per cent copper-iron alloy shows the change in susceptibility with increasing field strength. The susceptibility *versus* field

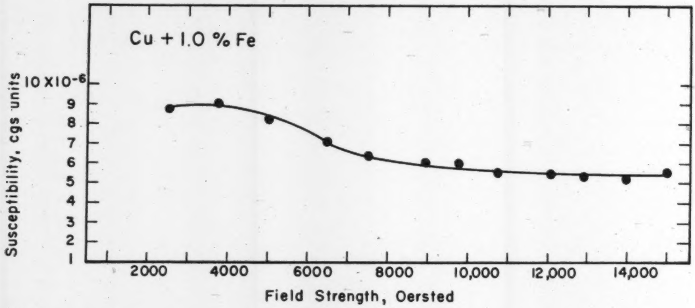


FIG. 10.—Susceptibility as a Function of Field Strength for a Copper - 1.0 per cent Iron Alloy Previous history: solution treated, cold worked 60 per cent, and aged 10 hr at 600 C.

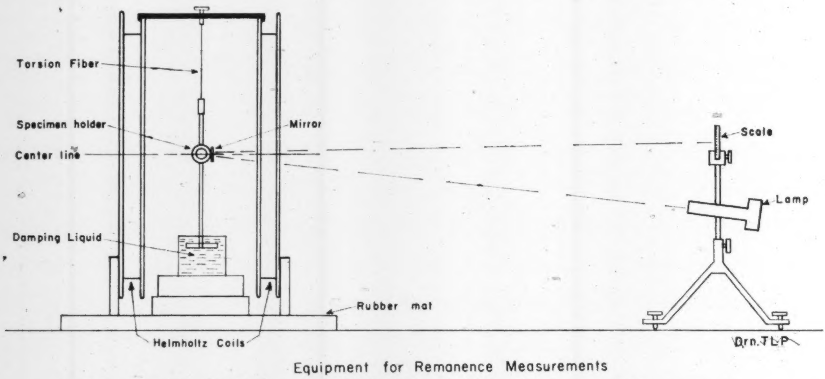

Torsion Fiber
Specimen holder
Mirror
Center line
Scale
Lamp
Damping Liquid
Rubber mat
Helmholtz Coils
Drn. TLP
Equipment for Remanence Measurements

FIG. 11.—Torsion Balance and Helmholtz Coils for Remanence Measurements.

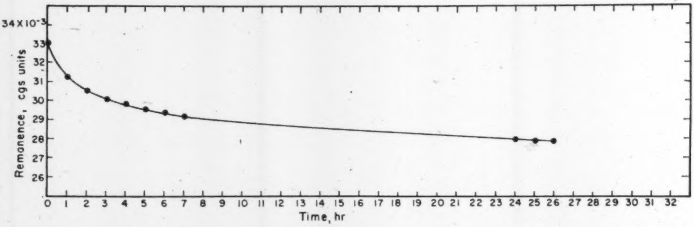


FIG. 12.—Remanence as a Function of Time After Magnetizing of 70-30 Brass with 0.34 per cent Iron. Solution treated and aged at 600 C for 24 hr.

strength curve reaches a maximum at saturation at about 3500 oersteds, then falls off slightly and becomes constant at higher fields. Measurements for this work were made at 15,000 oersteds with a gradient of 1500 oersteds per cm.

Remanence measurements were made 24 hr after magnetization in the uniform field of Helmholtz coils previously described and shown in Fig. 11. A deflection corresponding to a remanence of 10^{-5} could be seen. The maximum field used was 100 oersteds. The curve in Fig. 12 shows that remanence falls off rapidly at first but after 24 hr approaches a constant value.

Nine alloys were made for testing an iron series in copper, 70-30 alpha brass, and 52-48 beta brass. Analyses of these alloys are given in Table VIII.

TABLE VIII.—COMPOSITION OF ALLOYS USED IN TESTING AN IRON SERIES.

Number	Copper, per cent	Zinc, per cent	Iron, per cent	Tin, per cent	Aluminum, per cent	Lead, per cent
1	97.69	0.01	2.20	0.03	0.06	0.01
2	98.97	0.01	1.00	<0.01	0.02	<0.01
3	99.51	<0.01	0.47	<0.005	0.02	<0.01
4	99.76	0.10	0.14	<0.005	<0.01	<0.01
5	73.17	25.98	0.79	<0.01	0.06	0.01
6	71.82	27.70	0.43	<0.005	0.05	0.015
7	70.45	29.45	0.10	<0.005	<0.01	0.01
8	53.67	46.23	0.082	<0.003	...	0.018
9	52.23	48.18	0.048	<0.002	...	<0.01

Castings 1 in. in diameter were reduced to $\frac{1}{4}$ in. in diameter by cold swaging in the case of the coppers and 70-30 brasses with anneals as required and by forging and hot rolling in the case of the beta brasses.

Preliminary experiments with the copper-iron series indicated that despite efforts to obtain a rapid quench there was always some precipitation of iron with a wide scatter in magnetic properties. The use of a hydrogen atmosphere for heating and the increase of quenching temperature to 1030 C improved results. It was evident that some iron was being oxidized to a magnetically "hard" form, Fe_3O_4, and a high temperature was necessary to retain the iron in solution. All solution treatments for the copper-iron series were made by heating for 2 hr at 1030 C in an atmosphere of 93 per cent nitrogen - 7 per cent hydrogen and quenching in brine and ice.

The alpha brasses were solution treated at 850 C for 2 hr, the beta brasses for 1 hr, and water quenched. There was included in the original beta brass series an alloy of 0.5 per cent iron which was abandoned because the susceptibility could not be reduced to zero or even to a low value despite many quenching attempts. The iron in all these alloys (even the 0.5 per cent iron in beta brass) should be in solution at the quenching temperatures used.

Susceptibility and remanence were determined on every specimen after solution treating to be certain that no ferromagnetic precipitate was present before aging or working.

Aging temperatures were 400 and 600 C for various times up to 250 hr. In addition the copper-iron series and alpha brass - iron series were given cold reductions of 10, 30, and 60 per cent between solution and aging. To establish an aging curve the same specimen was used for the entire curve. The specimen was aged, determinations made on the specimen, then it was aged additional time, determinations again made, and so on until completion. This was to avoid discrepancies due to nonuniform specimens either from segregation or quenching effects. For short times up to 6 min aging was done in a salt bath and for longer times in a laboratory furnace with the specimens in a graphite container.

Specimens were quenched after aging. Aging temperatures were held to ±5 C.

Results and Discussion:

Typical aging curves of magnetic properties *versus* log time of aging are presented in the following. The first point shown on each curve represents the value before aging.

Copper Iron Series.—Susceptibility values for the 2.2 per cent iron alloy aged at 400 C (Fig. 13) show no aging effect except in the specimen worked 60 per cent before aging. In this specimen susceptibility increased immediately on working to 50×10^{-6} and then further increased quickly to 140×10^{-6} in 3 min aging. Since all solution treated specimens were first checked to be sure they had low magnetic values, it would seem that some iron was not in solution, that it was made magnetic first by working, then in short aging times, and that no further magnetic precipitation occurred at this temperature. The corresponding values for remanence fell off after short aging time with high values, 100×10^{-3}, for the worked specimens (Fig. 14).

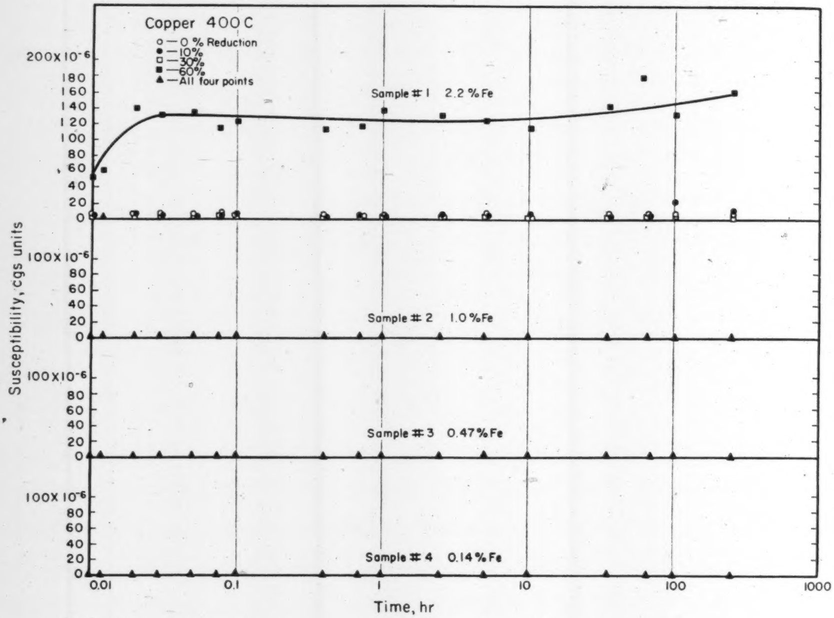


FIG. 13.—Susceptibility as a Function of Time for the Copper-Iron Series. Solution treated at 1030 C, aged at 400 C.

For the same 2.2 per cent iron alloy aged at 600 C (Fig. 15) it is shown that the worked specimens had low values for susceptibility, 30×10^{-6}, which increased rapidly with aging times over 10 hr to values up to 450×10^{-6}. The material not worked shows no aging effect, and worked specimens show most aging.

Remanence increases to high values with worked specimens but unworked specimens show no aging effect (Fig. 16).

The 1 per cent iron alloy aged at 400 C had very slight magnetic properties with some slight aging effect with time. The specimens cold worked 60 per cent showed the most response with remanence up to 50 × 10^{-3}, a low value (Figs. 13 and 14). Aged at 600 C there was slightly more response than at 400 C but still low values with remanence of 60 per cent cold worked specimens again showing the most response up to 190 × 10^{-3} (Figs. 15 and 16).

The two lower iron content alloys of 0.47 and 0.14 per cent show no aging effects at either 400 or 600 C except a slight increase in remanence, 30 × 10^{-3}, for 0.47 per cent iron specimen worked 60 per cent and aged 250 hr at 600 C (Figs. 13, 14, 15, and 16).

Microexamination of many of these specimens were made in both the solution treated and aged condition. In addition, observations were made using the method

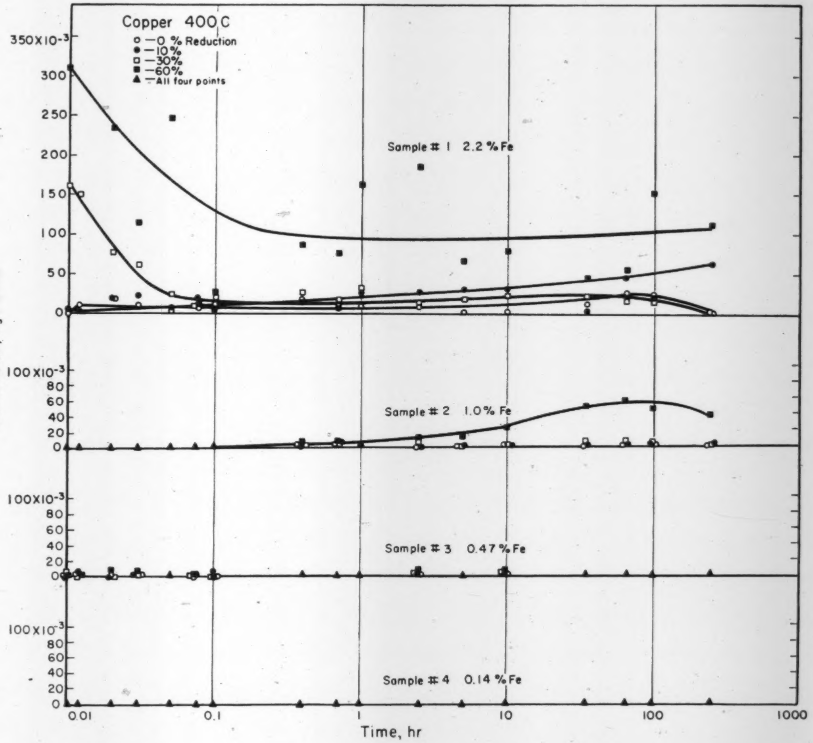


FIG. 14.—Remanence as a Function of Time for the Copper-Iron Series. Solution treated at 1030 C, aged at 400 C.

with colloidal suspensions of magnetite in a magnetic field as described by Avery, Homerberg, and Cook (12). Figures 17 and 18 are micrographs of typical specimens of the 2.2 per cent iron alloy in copper, solution treated, and solution treated and aged. The use of the colloid and magnetic field with the aged specimens shows the presence of the magnetic precipitate.

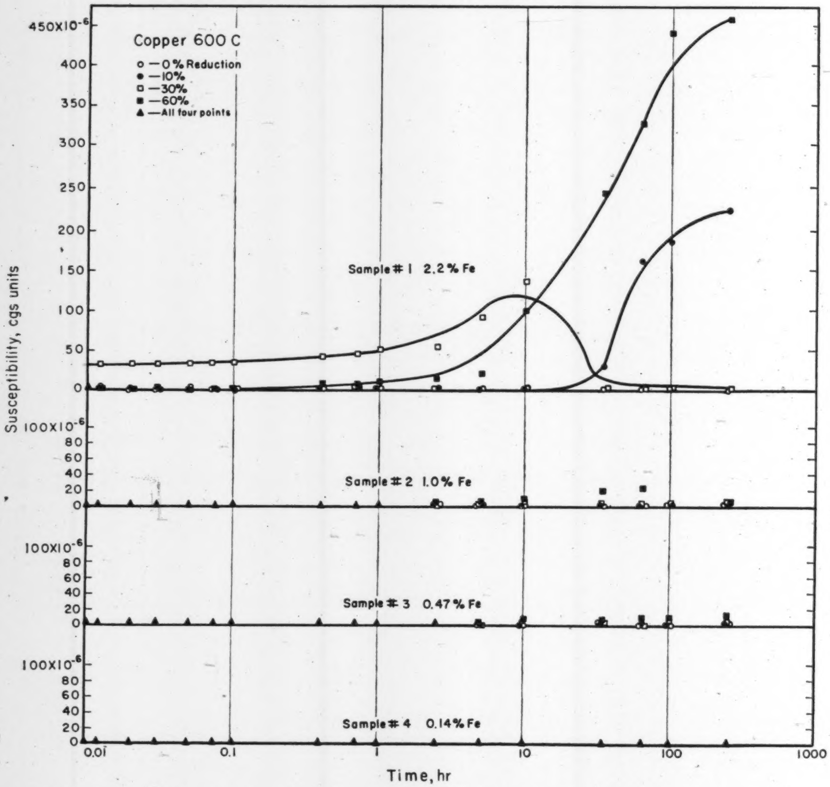


FIG. 15.—Susceptibility as a Function of Time for the Copper-Iron Series. Solution treated at 1030 C, aged at 600 C.

Alpha Brass Series.—The 0.79 per cent iron alloy aged at 400 C (Fig. 19), similar to the high iron 60 per cent cold worked in the copper series, shows susceptibility immediately in worked specimens but, contrary to the copper-iron alloy, the values increase slowly with long aging times to 300 to 500 × 10^{-6}. Specimens only solution treated show no aging effects. Remanence (Fig. 20) shows very little change with aging except specimens cold worked 30 per cent, and the specimens cold worked to 60 per cent have very high values, 1500 × 10^{-3}. The same

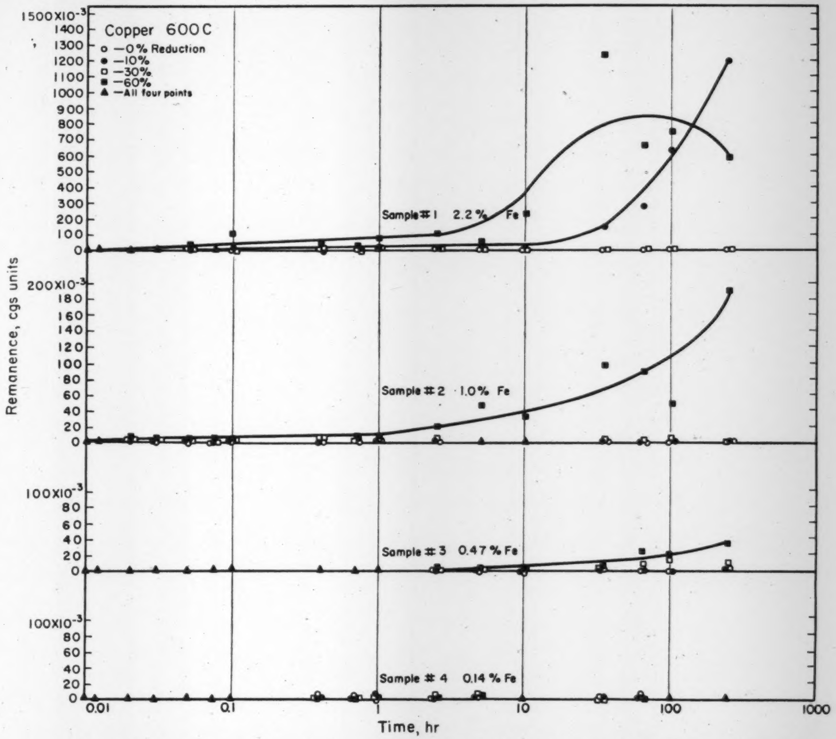


FIG. 16.—Remanence as a Function of Time for the Copper-Iron Series. Solution treated at 1030 C, aged at 400 C.

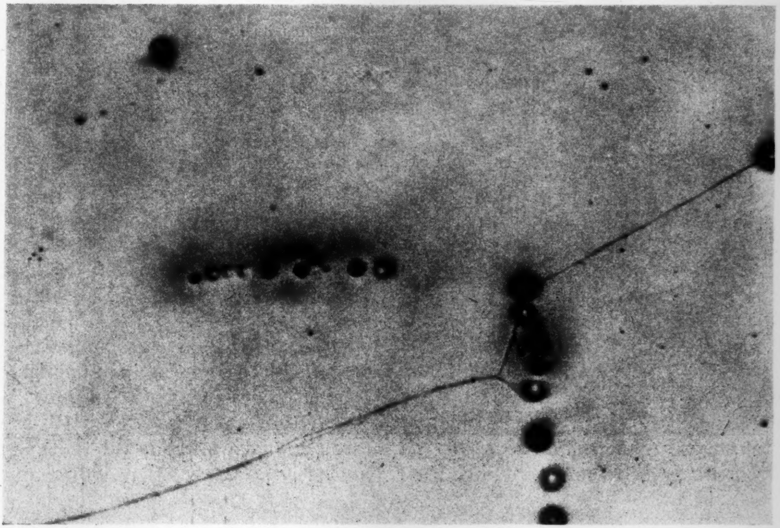
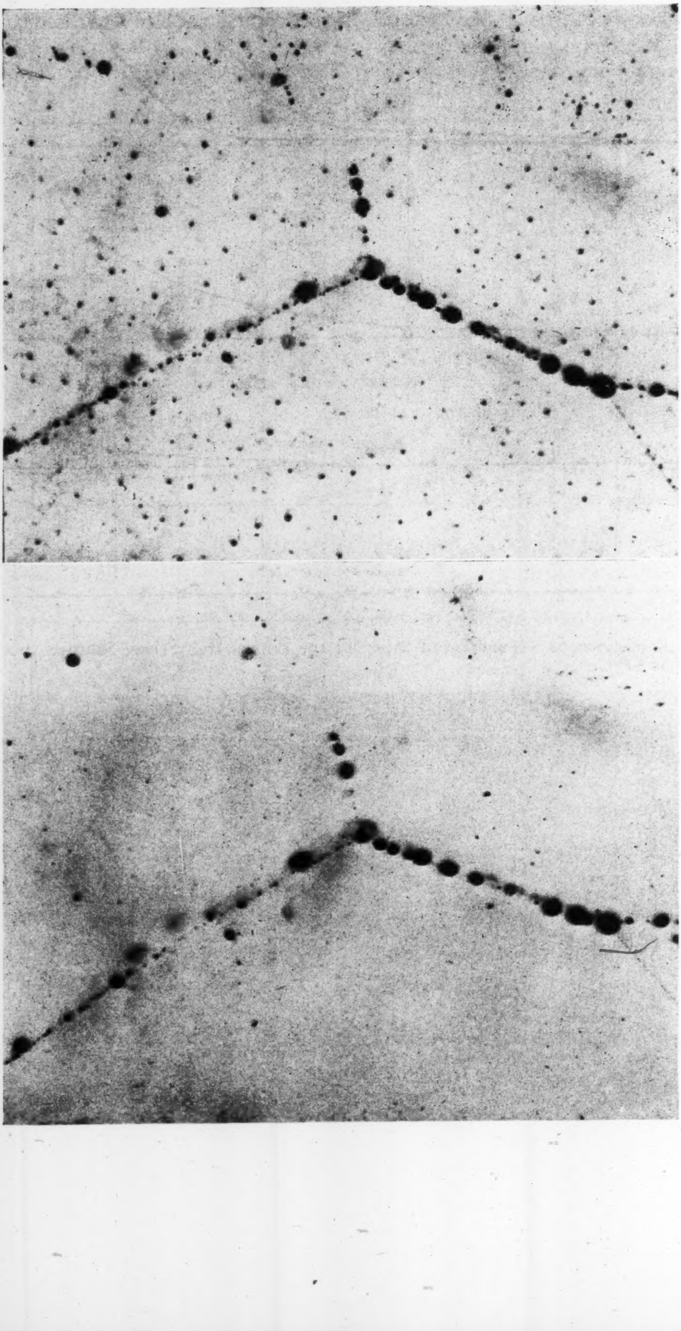


FIG. 17.—Copper - 2.2 per cent Iron Alloy Solution Treated at 1030 C. Electrolytically polished (×500).



(a) Without magnetic field.

(b) With magnetic field.

FIG. 18.—Copper - 2.2 per cent Iron Alloy Solution Treated at 1030 C and Aged at 600 C for 250 hr. Electrolytically polished and covered with a colloidal suspension of magnetite (×500).

alloy at 600 C (Fig. 21) changes rapidly with aging to high values of susceptibility for all conditions, both solution treated only and solution treated and worked. It shows aging effects to fairly constant values in a short time (1 hr) for specimens worked with heavy reductions. Specimens given light reductions and no reduction show aging effects later but with higher values. This is demonstrated by the crossing of these curves. Remanence shows very little aging effect (Fig. 22).

The 0.43 per cent iron alloy aged at 400 C (Fig. 19) shows a slight increase in susceptibility, 20×10^{-6}, with long aging times, 250 hr. The remanence (Fig. 20) shows aging at a greater rate, up to 400×10^{-3}, especially the worked specimens. The same iron content in copper (Figs. 13 and 14) shows no aging effect whatever at this temperature. Susceptibility increases rapidly with aging at 600 C (Fig 21) with the same crossing of the curves as noted in the 0.79 per cent iron alloy showing high values for light reductions. This effect is probably due to the particle size of the precipitated iron, the particles precipitated first on the heavily worked specimens being smaller than those precipitated later on the less

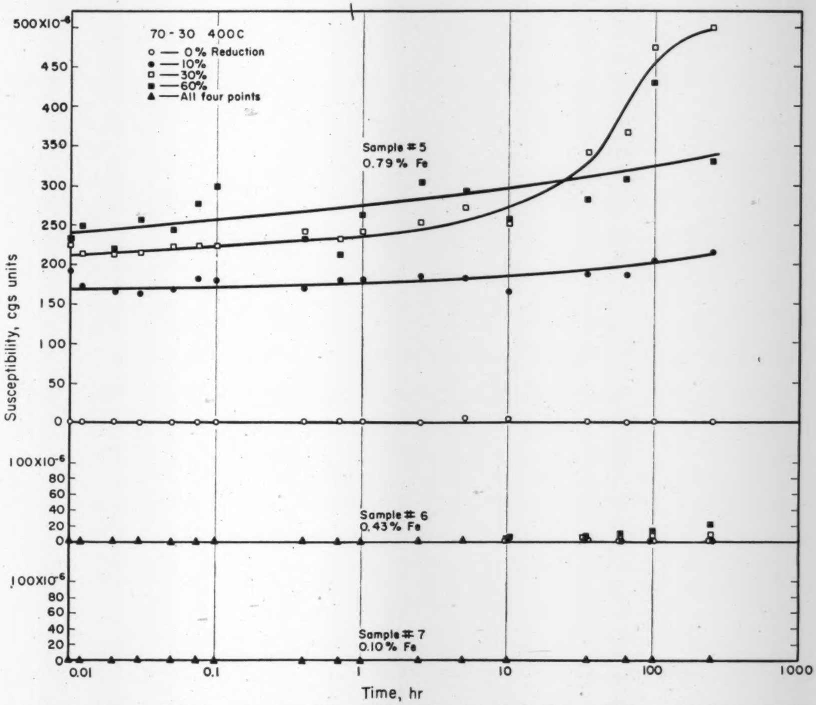


Fig. 19.—Susceptibility as a Function of Time for the Alpha Brass Series. Solution treated at 850 C, aged at 400 C.

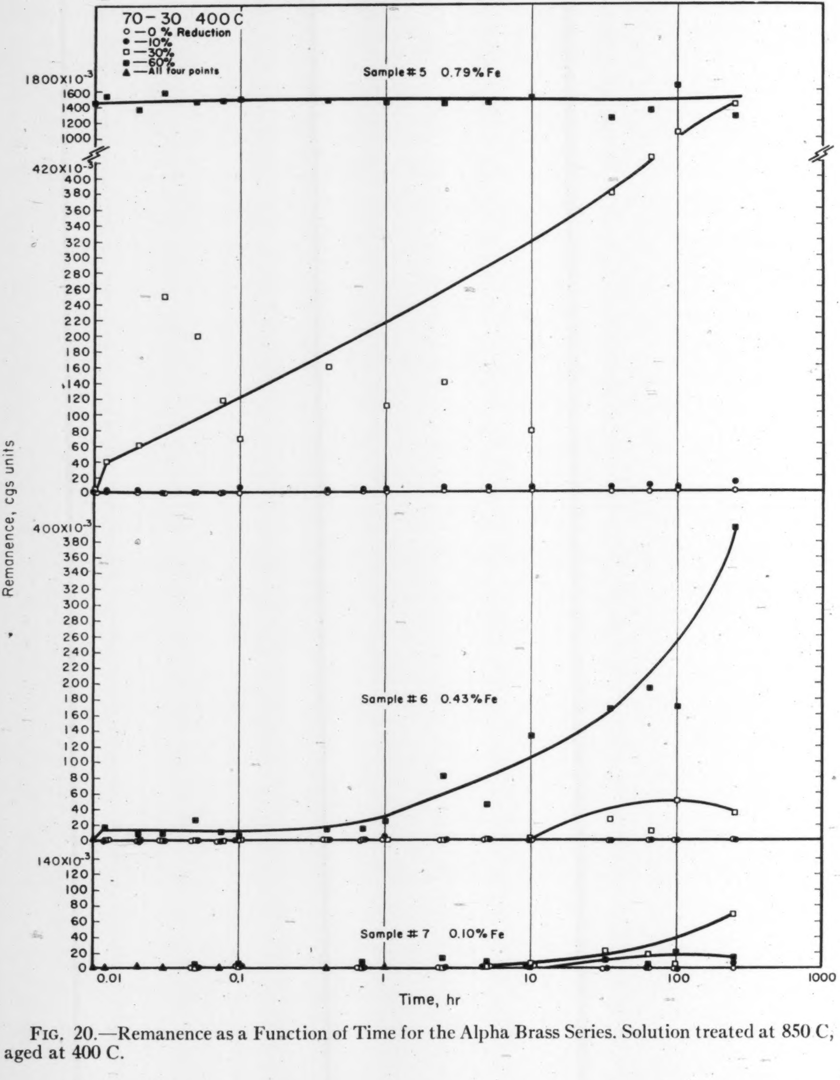


FIG. 20.—Remanence as a Function of Time for the Alpha Brass Series. Solution treated at 850 C, aged at 400 C.

everely worked specimens. Remanence follows the same pattern as susceptibility (Fig. 22).

The 0.1 per cent iron alloy has very low susceptibility with no effect of aging at either 400 or 600 C while remanence shows very slight aging effect up to 70 to 90×10^{-3} for both temperatures for long time, 250 hr (Figs. 19, 20, 21, and 22).

Micrographs of typical specimens solution treated and as aged are shown in Figs. 23 and 24. For aged specimens the colloidal suspension method was also used to show magnetic precipitation.

Aging both alloys at 400 C (Fig. 25) shows susceptibility increasing to maximum values of 118 × 10^{-6} in ½ hr and then falling off with longer times. These values are greater in the beta brass than in either alpha brass or copper for like amounts of iron. Remanence curves show a similar pattern (Fig. 26). Aging these alloys at 600 C showed no effect because this was in the solid solution range and no precipitation took place.

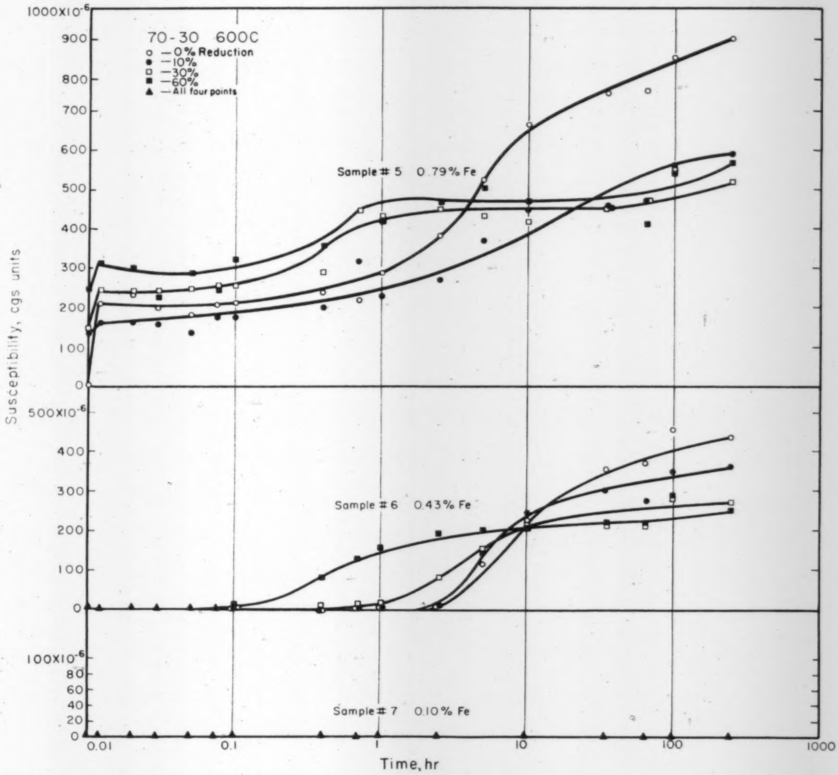


FIG. 21.—Susceptibility as a Function of Time for the Alpha Brass Series. Solution treated at 850 C, aged at 600 C.

Beta Brass.—Because of experimental difficulties with the beta brasses, aging experiments were made on only two alloys, 0.082 and 0.048 per cent iron. Original 0.5 per cent iron specimens could not be quenched to give low susceptibility values; 0.2 and 0.3 per cent iron specimens dezincified during forging, and alpha phase was observed in the microstructure.

Effect of Iron.—Increasing the amount of iron increases the susceptibility slowly at first and then at an increasing rate as

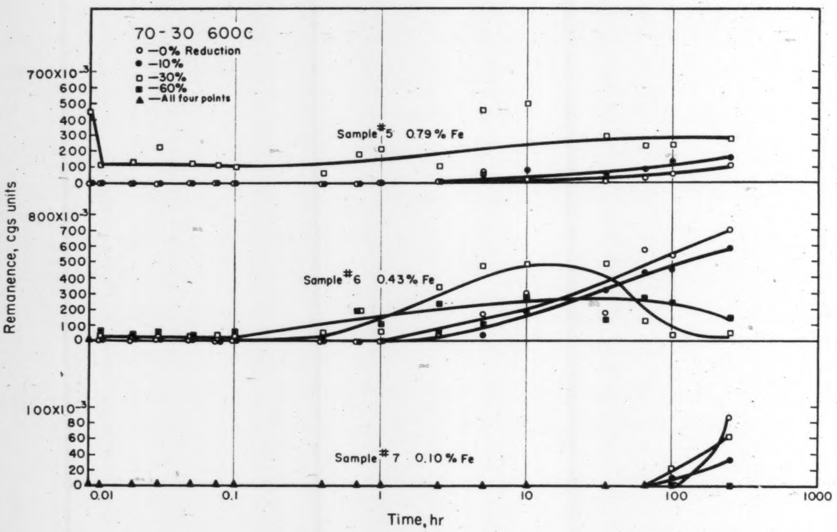


FIG. 22.—Remanence as a Function of Time for the Alpha Brass Series. Solution treated at 850 C, aged at 600 C.

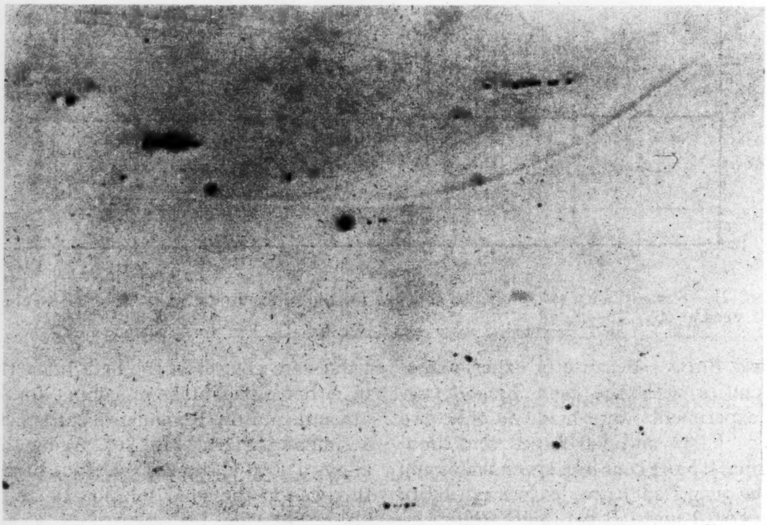
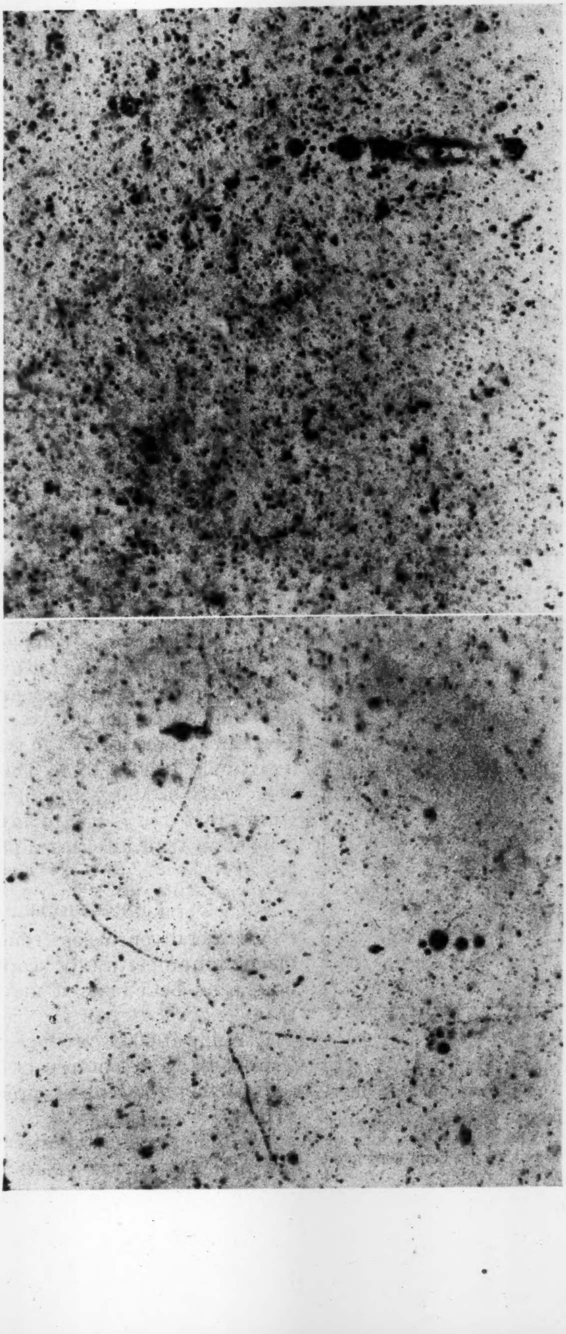


FIG. 23.—Iron, 0.79 per cent, in Alpha Brass. Solution treated at 850 C; electrolytically polished (×500).



(*a*) Without magnetic field. (*b*) With magnetic field.

FIG. 24.—Iron, 0.79 per cent, in Alpha Brass. Solution treated at 850 C; aged at 600 C for 250 hr. Electrolytically polished and covered with a colloidal suspension of magnetite (×500).

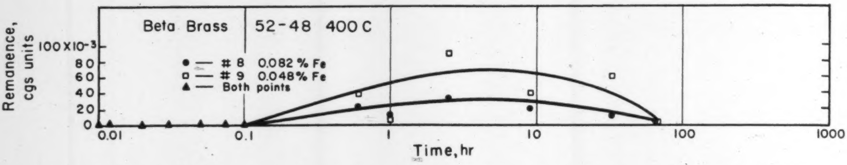


FIG. 25.—Susceptibility as a Function of Time for the Beta Brass Series. Solution treated at 850 C, aged at 400 C.

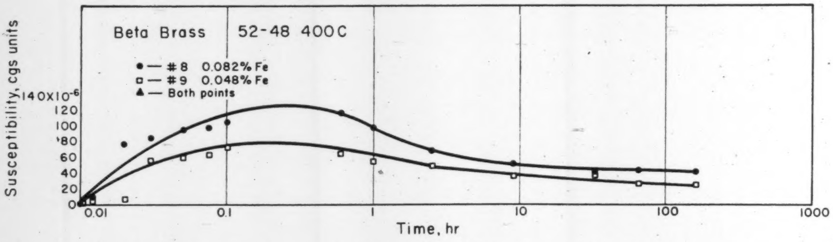


FIG. 26.—Remanence as a Function of Time for the Beta Brass Series. Solution treated at 850 C aged at 400 C.

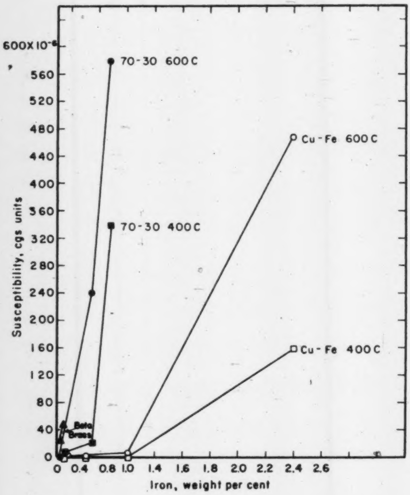


FIG. 27.—Susceptibility as a Function of Iron Content in Copper, Alpha Brass, and Beta Brass.

Copper solution treated at 1030 C, worked 60 per cent, and aged 250 hr. Alpha brass solution treated at 850 C, worked 60 per cent, and aged 250 hr. Beta brass solution treated at 850 C and aged 2.5 hr at 400 C.

shown in Fig. 27 for both copper and alpha brass. The relative position of these curves also demonstrates the great effect of iron on alpha brass compared to copper. For example, 0.5 per cent iron in alpha brass produces a remanence of 240 × 10^{-3} while in copper it is only 5 × 10^{-3}. The same comparison for 0.8 per cent iron would be 620 to 8. The curve for beta brass in this same figure demonstrates that iron is still more effective in beta brass than in either alpha brass or copper.

CONCLUSIONS

As general conclusions concerning the magnetic properties of copper, alpha brass, and beta brass it may be stated that:

1. Aging at 600 C produces more effect than at 400 C except in beta brass.
2. Higher iron contents produce higher values.
3. Aging these metals after cold working produces higher values than after solution treating only, except for alpha

brass with long aging time at 600 C where the reverse seems to be the case.

4. A given amount of iron produces a greater effect in beta brass than in alpha brass and the least effect in copper.

Acknowledgments:

The authors wish to acknowledge the helpful guidance and interest of Drs. D. K. Cramptom and H. L. Burghoff of the Research and Development Department of the Chase Brass & Copper Co., Inc., where this work was carried out; the assistance of H. Hughson for metallographic work; and the help of others in this department. Thanks are also extended to Dr. W. D. Robertson of Yale University for counsel during the work. The samples of aluminum bronze were furnished by Ampco Metal, Inc.

REFERENCES

(1) Allison Butts and Paul L. Rieber, Jr., "Magnetism in Copper Alloys; Effect of Iron as Impurity," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 857 (1949).

(2) F. Bitter and A. R. Kaufman, "Magnetic Studies of Solid Solutions. I. Methods of Observation and Preliminary Results on the Precipitation of Iron from Copper," *The Physical Review*, Vol. 56, p. 1044 (1939).

(3) R. B. Gordon and M. Cohen, "Age Hardening of a Copper-Cobalt and a Copper-Iron Alloy," "Age Hardening of Metals," Am. Soc. Metals (1939).

(4) C. S. Smith, "Structure and Ferromagnetism of Cold-Worked Copper Containing Iron," *The Physical Review*, Vol. 57, p. 335 (1940).

(5) F. Bitter, A. R. Kaufman, C. Starr, and S. T. Pan, "Magnetic Studies of Solid Solutions. II. The Properties of Quenched Copper-Iron Alloys," *The Physical Review*, Vol. 60, p. 134 (1941).

(6) C. S. Smith, "Magnetic Studies on the Precipitation of Iron in Alpha and Beta Brass," *Transactions*, Am. Inst. Mining, Metallurgical and Petroleum Engrs., Vol. 147, p. 111 (1942).

(7) F. W. Constant, R. E. Faires, and H. E. Lenander, "Investigation of Ferromagnetic Impurities. II." *The Physical Review*, Vol. 63, p. 441 (1943).

(8) O. Bauer and M. Hansen, "Der Einflusz von dritten metallen auf die Konstitution der messinglegierungen, VI. Der Einflusz von Eisen," *Zeitschrift für Metallkunde*, Vol. 26, p. 121 (1934).

(9) R. E. Cech and D. Turnbull, "Magnetic Transformation of Iron in a Copper Matrix at Low Temperatures," *Journal of Metals*, Vol. 6, p. 45 (1954).

(10) M. R. Gross, "Magnetic Permeability of So-Called Non-Magnetic Metallic Materials," *Journal*, Am. Soc. Naval Engrs., Inc., Vol. 66, No. 1, p. 215 (1954).

(11) R. B. Sosman and J. B. Austin, "An Apparatus for Measuring the Magnetic Susceptibility of Liquids and Solids at High Temperatures," *Journal*, Washington Academy of Sciences, Vol. 25, p. 15 (1935).

(12) H. S. Avery, V. O. Homesberg, and Earnshaw Cook," "Metallographic Identification of Ferro Magnetic Phases," *Metals & Alloys*, Vol. 10, p. 353 (1939).

REPORT OF COMMITTEE B-6
ON
DIE-CAST METALS AND ALLOYS*

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: in Boston, Mass., on June 24, 1958, and in Pittsburgh, Pa., on February 3, 1959.

The committee consists of 87 members, of whom 43 are classified as producers, 19 as consumers, and 25 as general interest members.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Zinc-Base Alloy Die Castings (**B 86 – 57 T**), and Zinc-Base Alloys in Ingot Form for Die Castings (**B 240 – 57 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (E. V. Blackmun, chairman) reported a continuation of the study of determining mechanical properties for aluminum alloy G8A.

A specification change is now being balloted in the subcommittee, in which the permissible zinc content of alloys SC84A and SC84B will be increased to a maximum of 3.0 per cent to recognize the commercial usage of the high zinc version of the SC84 alloy.

Subcommittee II on Zinc-Base Die-Casting Alloys (A. E. Weiss, chairman) reported progress on the study of the mechanical properties of zinc alloys cast in evacuated dies in comparison with the properties of regularly produced die-cast test bars. Three die-casters have produced the test specimens and two of the lots have been tested.

Subcommittee V on Exposure and Corrosion Tests (D. H. Kleppinger, chairman) reported that the plans have been completed for the recalling and testing of the last of the zinc-alloy test bars of alloys XXI, XXIII, and XXV and the last of the magnesium-alloy bars of alloys 307, 312, 313, and 314 from each of seven sites after 20 yr of atmospheric exposure.

The program for the testing of the 6-yr outdoor aged die-cast bars of alloy SC84 with several zinc contents has also been arranged.

This report has been submitted to letter ballot of the committee, which consists of 87 members; 81 members returned their ballots, of whom 73 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. BABINGTON,
Chairman.

GEORGE L. WERLEY,
Secretary.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT*

Committee B-7 on Light Metals and Alloys, Cast and Wrought, held two meetings during the year: at Boston, Mass., in June 1958, and at Pittsburgh, Pa., in February 1959.

The committee consists of 90 members of whom 87 are voting members; 37 are classified as producers, 31 as consumers, and 22 as general interest members.

R. A. Quadt resigned as chairman of Subcommittee I and was replaced by D. L. LaVelle. R. T. Wood passed away and F. L. Chase resigned from the committee because of retirement.

The Advisory Committee continued to function as advisor to the American Standards Assn. in its representation of the United States as a participating member of Technical Committee 79 on Light Metals and Their Alloys of the International Organization for Standardization (ISO). L. H. Adam attended the June 1958 meeting of ISO/TC79 at Harrogate, England as the United States representative.

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends one new specification for publication as tentative, revisions in 17 tentative specifications, and revision of 10 standards for immediate adoption. The committee also recommends that each standard and tentative covering light metals and alloys indicate the subcommittee having jurisdiction over it. The standards and tentatives affected, together with the revisions recommended, are covered in detail in Appendix I to this report.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum Alloy Ingots (D. L. LaVelle, chairman) revised the Standard Specification for Aluminum for Use in Iron and Steel Manufacture (B 37 – 57) to delete reference to specifying inspection at the point at which material is received. The subcommittee also revised the Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 58) to add alloy 995A with conductivity requirements; to delete alloys SC64A and SC64B, and add alloy SC64D to replace them; and to delete reference to specifying inspection at the point at which material is received. The revisions in these specifications are set forth in Appendix I to this report.

Subcommittee II on Aluminum Alloy Castings (D. L. Colwell, chairman) revised the Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 – 58 T) to add the additional tempers F and T71 for alloy SG70A; to delete the higher temperature artificial aging treatment and add a melting range for alloy ZC81A; and to delete reference to specifying inspection at the point at which material is received. The subcommittee also revised the Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 58 T) to add new alloys SC51B and SG70B (the high-purity grades of alloys SC51A and SG70A) in the T61 temper, and the additional tempers F and T71 for SG70A; to add a melting range for alloy ZC81B; and to delete reference to specifying inspection at the point at which material is received. The revisions in these specifications are in Appendix I to this report.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] See p. 267.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The subcommittee cooperated in the development of ISO Recommendation No. 258 on Definition and Classification of Composition of Aluminum Alloy Castings, which is now being balloted on by ISO/TC79.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (H. D. Monsch, chairman) prepared the new Tentative Specification for Aluminum-Alloy Pipe for Gas and Oil Transmission and Distribution Piping Systems.

The subcommittee revised Tentative Specification B 209 to delete the beryllium limit and reduce the copper limit for alloy GM51A (5456); to add the 0, H32, and H34 tempers for alloy GM41A (5083) sheet; to correct the elongation and tensile strength for Clad CS41A-0 alloy (Alclad 2014-0); to add minimum yield strength requirements for alloys M1A-H16 (3003-H16), MG11A-H36 (3004-H36), G1A-H36 (5050-H36), and GR20A-H36 (5052-H36); to revise some thickness increments for tensile requirements; to exempt the manufacturer from conducting bend tests; and to delete reference to specifying inspection at the point at which material is received.

Specification B 210 was revised to add the H16 temper for alloy M1A (3003), and the H36 temper for alloys MG11A (3004), G1A (5050), and GR20A (5052), all with minimum yield strength requirements; to add minimum wall thickness dimensions for tensile requirements for some alloys and tempers; and to delete reference to specifying inspection at the point at which material is received.

Specification B211 was revised to add alloy M1A (3003) in the 0 and H112 tempers, and alloy GR40A (5154) in the 0, H112, and F tempers; and to delete reference to specifying inspection at the point at which material is received.

Specifications B 221, B 235, and B 308 were revised to delete the beryllium limit and reduce the copper limit for alloy GM51A (5456); and to delete reference to specifying inspection at the point at which material is received.

Changes in Specification B 241 were made to delete the beryllium limit and reduce the copper limit for alloy GM51A (5456); to revise the outside diameter and wall thickness tolerances for Schedules 5 and 10 pipe; and to delete reference to specifying inspection at the point at which material is received.

Changes in Specifications B 234, B 236, B 247, B 307, B 313, B 316, B 317, and B 318 were made to delete reference to specifying inspection at the point at which material is received.

The subcommittee has a task group developing a tentative specification for aluminum foil for capacitors, and a task group working on a major revision of the Tentative Specification for Extruded Aluminum-Alloy Bars, Rods, Pipe, and Structural Shapes for Electrical Purposes (B 317).

The subcommittee assisted in the development of a new Tentative Specification for Aluminum-Alloy Welding Fittings as a participating member of a special coordinating committee comprising representatives of Committees B-2, B-5, and B-7.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (A. A. Moore, chairman) revised Specification B 80 to add new alloy K1A in the F temper, and to delete reference to specifying inspection at the point at which material is received.

Revisions of Specification B 90 were made to add new alloys ZE10A in the 0 and H24 tempers, and HM21A in the T8 temper; to add limits for zinc, copper, and nickel for alloy HK31A; to revise the tensile requirements for AZ31B and add tensile requirements for HK31A plate; and to delete reference to specifying inspection at the point at which material is received.

Revisions of Specifications B 91, B 93, B 107, B 199, and B 217 were made to delete reference to specifying inspection at the point at which material is received.

A task group is considering composition limits for high purity magnesium for nuclear applications.

Subcommittee V on Testing Light Metals (J. C. Millson, chairman), through having representatives on the Test Methods Panel of the Joint ASTM-ASME Committee on the Effect of Temperature on the Properties of Metals, has been instrumental in having the Tentative Recommended Practice for Short-Time Elevated-Temperature Tension Tests of Materials (E 21) revised to add strain rates.

The subcommittee has been unable to obtain agreement on expressing stress values in terms of thousands of pounds per square inch in B-7 specifications, and no progress has been made in developing sampling requirements based on statistical methods because Committee E-11 on Quality Control of Materials has been unable to formulate recommendations.

A task group has recommended the following minimum dimensions for round and flat subsize tension test specimens for cast and wrought light metals and alloys:

Round specimens:

Reduced section, ¼ in. diameter by 1 in. gage length;
Grip ends, ⅜ in. diameter;
Total length, 3 in. if tested with threaded or shouldered ends, or 4 in. if tested with wedge grips.

Flat specimens:

Reduced section, ¼ in. wide by 1 in. gage length;
Grip ends, ⅜ in. wide;
Total length, 4 in.

Subcommittee VI on Anodic Oxidation of Aluminum and Magnesium Alloys (R. F. Hafer, chairman) is revising Standard Methods of Measuring Thickness of Anodic Coatings on Aluminum by Means of the Filmeter (B 244 - 56) to include other film measuring instruments, and has completed a proposed tentative specification for anodic coatings on aluminum and aluminum alloys with the exception of an appendix.

A paper by G. E. Best, J. G. Hecker, J. W. McGrew, and R. V. VandenBerg on "Chromic Acid Anodizing Characteristics of Wrought Aluminum Alloys"[3] was presented at the February 1959 Meeting.

Subcommittee VII on Codification of Light Metals and Alloys, Cast and Wrought (C. B. Gleason, chairman) revised the Standard Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 - 58) to delete reference to Tentative Recommended Practice for Designating Significant Places in Specified Limiting Values (E 29) because the 1958 revision no longer contains the rounding-off method used in establishing ASTM designations for light metals and alloys in accordance with Recommended Practice B 275. The revision of B 275 adds the rounding-off method formerly in Recommended Practice E 29.

---

[3] See p. 277.

Subcommittee VIII on Atmospheric Exposure Tests (L. H. Adam, chairman) has test specimens of aluminum and magnesium alloys, cast and wrought, at five ASTM atmospheric exposure test sites: on top of the Port Authority Building, New York City, for industrial atmospheric conditions; at State College, Pa., for rural; at Kure Beach, N. C., for Atlantic Coast marine; at Point Reyes, Calif., for Pacific Coast marine; and at Freeport, Tex., for Gulf Coast marine atmospheric conditions. The data on exposure for six months, one year, and three years are complete. The specimens exposed for five years at New York City, State College, and Kure Beach are being tested, and those at Point Reyes and Freeport are being removed for testing. Data obtained from wrought aluminum and magnesium alloys exposed for three years are given in Appendix III to this report.[4]

Subcommittee IX on Editorial (R. A. Harris, chairman) is preparing Recommendations on Form of ASTM Committee B-7 Specifications to supplement Proposed Recommendations on Form of ASTM Specifications, April 1957, which have been approved as a guide, insofar as practical, for writing and revising B-7 specifications. Standardization of section titles, sequence, and wording, and form, titles, and headings of tables is being attempted.

Special Subcommittee on Light Metals for Nuclear Applications (W. G. Groth, chairman) is cooperating with Subcommittee IV in developing requirements for high-purity magnesium in ingot form of a quality suitable for use as a reducing agent in the production of nuclear grade metals.

This report has been submitted to letter ballot of the committee, which consists of 90 members; 54½ members returned their ballots, of whom 51½ have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

I. V. WILLIAMS,
Chairman.

R. B. SMITH,
Secretary.

---

[4] See p. 289.

APPENDIX I

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON LIGHT METALS AND ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards and tentatives covering light metals and alloys which are referred to earlier in this report. The standards and tentatives appear in their present form in the 1958 Book of ASTM Standards, Part 2.

NEW TENTATIVE

The committee recommends for publication as tentative the Specification for Aluminum-Alloy Pipe for Gas and Oil Transmission and Distribution Piping Systems as appended hereto.[1]

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 – 58 T):

Section 2.—Revise Item (*6*) to read as follows: "(*6*) Whether inspection is required at the manufacturer's works (Section 14(*a*))."

Table II.—Add the additional tempers F and T71 for alloy SG70A with the following tensile requirements:

	F (As Cast)	T71 (Solution Treated and Overaged)
Yield strength, min, psi.	. . .	18 000
Tensile strength, min, psi	19 000	25 000
Elongation in 2 in., min, per cent	2.0	3.0

Table III.—For ZC81A delete the aging treatment of "1 day at room temperature + 310 ± 5 F for 9 hr."

Section 14(a).—Revise to read as follows:

> 14. (*a*) If the purchaser desires to make an inspection of the castings at the manufacturer's works where the castings are made, it shall be so stated in the contract or order.

Table IV.—For alloy ZC81A add an approximate melting range of "1100 to 1185 F."

Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 58 T):

Section 2.—Revise Item (*6*) to read as follows: "(*6*) Whether inspection is required at the manufacturer's works (Section 14(*a*))."

Table I.—Add alloys SC51B and SG70B with the chemical requirements shown in the accompanying Table I.

Table II.—Add alloys SC51B and SG70B in the T61 temper, and the additional tempers F and T71 for SG70A, with the tensile requirements shown in the accompanying Table II.

Section 14(a).—Revise as indicated above for Section 14(*a*) in Tentative Specification B 26.

Table IV.—Add an approximate melting range of "1100 to 1185 F" for alloy ZC81B, and add alloys SC51B and SG70B with the properties and characteristics shown in the accompanying Table III.

---

[1] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 2.

TABLE I.—ADDITIONS TO TABLE I, SPECIFICATION B 108 - 58 T.

	SC51B	SG70B
Aluminum, per cent	remainder	remainder
Copper, per cent	1.0 to 1.5	0.20
Iron, per cent	0.20	0.20
Silicon, per cent	4.5 to 5.5	6.5 to 7.5
Manganese, per cent	0.10	0.10
Magnesium, per cent	0.40 to 0.6	0.20 to 0.40
Zinc, per cent	0.10	0.10
Titanium, per cent	0.20	0.20
Other elements, per cent:		
Each	0.05	0.05
Total	0.15	0.15

Table II.—Revise the tensile requirements for Clad CS41A-0 (Alclad 2014-0) to correct the elongation and tensile strength requirements; for M1A-H16 (3003-H16), MG11A-H36 (3004-H36), G1A-H36 (5050-H36), and GR20A-H36 (5052-H36) to add minimum yield strength requirements; and for GM41A (5083) to add the 0, H32, and H34 tempers for sheet; as shown in the accompanying Table IV.

Section 18(a).—Revise to read as follows:

> 18. (*a*) If the purchaser desires to make an inspection of the material at the manufacturer's works where the material is made, it shall be so stated in the contract or purchase order.

TABLE II.—ADDITIONS TO TABLE II, SPECIFICATION B 108 - 58 T.

Alloy	Condition	Yield Strength, min, psi	Tensile Strength, min, psi	Elongation in 2 in., min, per cent
SC51B	T61 (Solution treated and aged)	30 000	40 000	3.0
SG70A	F (As cast)	...	21 000	3.0
	T71 (Solution treated and over-aged)	...	25 000	3.0
SG70B	T61 (Solution treated and aged)	26 000	38 000	5.0

Tentative Specification for Aluminum-Alloy Sheet and Plate (B 209 - 58 T):

Section 2.—Revise Item (*8*) to read as follows: "(*8*) Whether inspection is required at the manufacturer's works (Section 18(*a*)).

Table I.—For alloy GM51A (5456) delete the beryllium limit and revise the present maximum copper limit of "0.20" to read "0.10" per cent.

Section 8.—Revise the first sentence to read "When bend requirements are specified on the purchase order, sheet and plate shall be capable of being bent cold through an angle of 180 deg around a pin having a diameter equal to *N* times the thickness of the sheet or plate without cracking, the value of *N* being as prescribed in Table II for the different alloys, tempers, and thicknesses, but the manufacturer need not conduct the test."

TABLE III.—ADDITIONS TO TABLE IV, SPECIFICATION B 108.

	SC51B	SG70B
Approximate melting range, deg Fahr	1015 to 1150	1035 to 1135
Foundry characteristics:		
Resistance to hot cracking	1	1
Pressure tightness	1	1
Fluidity	2	2
Solidification shrinkage tendency	2	1
Normally heat-treated	Yes	Yes
Other characteristics:		
Resistance to corrosion	3	2
Machining	3	3
Polishing	3	3
Electroplating	2	1
Anodizing (appearance)	4	4
Chemical oxide coating (protection)	2	2
Strength at elevated temperature	2	3
Suitability for welding	2	2
Suitable for brazing	No	No

TABLE IV.—REVISIONS OF TABLE II, SPECIFICATION B 209.

Temper	Thickness, in.	Tensile Strength, psi		Yield Strength, psi		Elongation in 2 in., min, per cent	Bend Diameter Factor, *N*
		Minimum	Maximum	Minimum	Maximum		
ASTM Alloy Clad CS41A or AA Alloy Alclad 2014							
0..........	0.020 to 0.499	...	30 000	...	14 000	16	...
	0.500 to 1.000	...	32 000	...	...	10	...
ASTM Alloy G1A or AA Alloy 5050							
H36.......	0.008 to 0.019	27 000	33 000	22 000[h]	...	2	3
	0.020 to 0.050	27 000	33 000	22 000[h]	...	3	3
	0.051 to 0.162	27 000	33 000	22 000[h]	...	4	4
ASTM Alloy GM41A or AA Alloy 5083							
0..........	0.050 to 0.750	40 000	...	18 000[h]	...	16	...
	0.751 to 1.500	38 000	...	16 000[h]	...	16	...
H32.......	0.050 to 0.125	45 000	52 000	34 000[h]	...	8	...
	0.126 to 0.249	45 000	52 000	34 000[h]	...	10	...
H34.......	0.050 to 0.125	50 000	57 000	39 000[h]	...	6	...
	0.126 to 0.249	50 000	57 000	39 000[h]	...	8	...
H113.......	0.250 to 2.000	44 000	51 000	31 000[h]	41 000[h]	12	...
ASTM Alloy GR20A or AA Alloy 5052							
H36.......	0.006 to 0.007	37 000	44 000	29 000[h]	...	...	...
	0.008 to 0.031	37 000	44 000	29 000[h]	...	3	4
	0.032 to 0.162	37 000	44 000	29 000[h]	...	4	5
ASTM Alloy M1A or AA Alloy 3003							
H16.......	0.006 to 0.019	24 000	30 000	21 000[h]	...	1	4
	0.020 to 0.031	24 000	30 000	21 000[h]	...	2	4
	0.032 to 0.050	24 000	30 000	21 000[h]	...	3	4
	0.051 to 0.162	24 000	30 000	21 000[h]	...	4	6
ASTM Alloy MG11A or AA Alloy 3004							
H36.......	0.006 to 0.007	35 000	41 000	28 000[h]	...	...	...
	0.008 to 0.019	35 000	41 000	28 000[h]	...	1	6
	0.020 to 0.031	35 000	41 000	28 000[h]	...	2	6
	0.032 to 0.050	35 000	41 000	28 000[h]	...	3	6
	0.051 to 0.162	35 000	41 000	28 000[h]	...	4	8

Tentative Specification for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 58 T):

Section 2.—Revise Item (7) to read as follows: "(7) Whether inspection is required at the manufacturer's works (Section 17(*a*))."

Table II.—Add tensile requirements for G1A-H36 (5050-H36), GR20A-H36 (5052-H36), M1A-H16 (3003-H16), and

TABLE V.—ADDITIONS TO TABLE II, SPECIFICATION B 210.

Alloy		Temper	Wall Thickness, in.	Tensile Strength, psi		Yield Strength, min, psi[a]	Elongation in 2 in. or 4 × Diameter, min, per cent[f]	
ASTM	AA			Minimum	Maximum		Full-Section Specimen	Cut-out Specimen
G1A	5050	H36	0.010 to 0.500	27 000	...	22 000[c]	...	...
GR20A	5052	H36	0.010 to 0.450	37 000	...	29 000[c]	...	...
M1A	3003	H16	0.010 to 0.500	24 000	...	21 000[c]	...	...
MG11A	3004	H36	0.018 to 0.450	35 000	...	28 000[c]	...	...

TABLE VI.—REVISIONS OF TABLE II, SPECIFICATION B 210.

Alloy		Temper	Wall Thickness, in.
ASTM	AA		
996A	1060	0	0.010 to 0.500
		H14	0.010 to 0.500
		H18	0.010 to 0.500
		H112	0.010 to 0.500
CG42A	2024	0	0.018 to 0.500
GS10A	6063	0	0.018 to 0.500
		T83	0.025 to 0.259
		T831	0.025 to 0.259
		T832	0.025 to 0.259
GS11A	6061	0	0.018 to 0.500
GS11C	6062	0	0.018 to 0.500
M1A	3003	0	0.010 to 0.500
		H14	0.010 to 0.500
		H18	0.010 to 0.500
Clad M1A	Alclad 3003	0	0.010 to 0.500
		H14	0.010 to 0.500
		H18	0.010 to 0.500
		H112	0.050 to 0.500
MG11A	3004	H34	0.018 to 0.450
		H38	0.018 to 0.450

MG11A-H36 (3004-H36) to read as shown in the accompanying Table V.

Revise wall thicknesses for the following alloys and tempers to read as shown in the accompanying Table VI.

Section 17(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Bars, Rods, and Wire (B 211 – 58 T):

Table I.—Add alloys GR40A (5154) and M1A (3003) with the chemical requirements as shown in the accompanying Table VII.

TABLE VII.—ADDITIONS TO TABLE I, SPECIFICATION B 211.

	ASTM GR40A (AA 5154)	ASTM M1A (AA 3003)
Aluminum, per cent	remainder	remainder
Copper, per cent	0.10	0.20
Iron, per cent	[d]	0.7
Silicon, per cent	[d]	0.6
Manganese, per cent	0.10	1.0 to 1.5
Magnesium, per cent	3.1 to 3.9	....
Zinc, per cent	0.20	0.10
Chromium, per cent	0.15 to 0.35	....
Titanium, per cent	0.20	....
Other elements, per cent		
Each	0.05	0.05
Total	0.15	0.15

TABLE VIII.—ADDITIONS TO TABLE II, SPECIFICATION B 211.

Alloy		Temper	Diameter or Thickness, in.	Tensile Strength, psi		Yield Strength, psi[a]	Elongation in 2 in. or 4 × Diameter, min, per cent[c]
ASTM	AA			Minimum	Maximum		
M1A	3003	O	All	14 000	19 000	5 000 min[b]	...
		H112	All	14 000	...	5 000 min[b]	...
		F	All	[d]	...	[d]	...
GR40A	5154	O	All	30 000	41 000	11 000 min[b]	...
		H112	All	30 000	...	11 000 min[b]	...

Section 2.—Revise Item (*8*) to read as follows: "(*8*) Whether inspection is required at the manufacturer's works (Section 17(*a*))."

Table II.—Add alloys M1A (3003) and GR40A (5154) with the tensile requirements shown in the accompanying Table VIII.

Section 7.—Revise the second sentence to read "Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of ASTM alloys 990A, 996A, GR20A, GR20B, GR40A, and M1A, yield strength is not important."

Section 17(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 – 58 T):

Section 2.—Revise Item (*8*) to read as follows: "(*8*) Whether inspection is required at the manufacturer's works (Section 16(*a*))."

Table I.—For alloy GM51A (5456) delete the beryllium limit and revise the present maximum copper limit of "0.20" to read "0.10" per cent.

Section 16(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 – 58 T):

Section 2.—Revise Item (*8*) to read as follows: "(*8*) Whether inspection is required at the manufacturer's works (Section 19(*a*)).

Section 19(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Extruded Tubes (B 235 – 58 T):

Section 2.—Revise Item (*8*) to read as follows: "(*8*) Whether inspection is required at the manufacturer's works (Section 17(*a*))."

Table I.—For alloy GM51A (5456) revise the present maximum copper limit of "0.20" to read "0.10" per cent, and delete footnote *g* and references to it.

Section 17(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

TABLE IX.—PERMISSIBLE VARIATIONS IN OUTSIDE DIAMETER.
(Revision of Table IV, Specification B 241)

Pipe Size, in.	Permissible Variations in Mean[a] Outside Diameter from the Nominal Diameter,[b] in.	Permissible Variations in Outside Diameter at Any Point from the Nominal Outside Diameter,[b] in.
	Schedule 5 and 10	Schedule 20 and Greater
Under 2...	+0.015, −0.031	+0.015, −0.031
2 to 4.....	+0.031, −0.031	±1 per cent
4½ to 7...	+0.062, −0.031	±1 per cent
8 to 12....	+0.093, −0.031	±1 per cent

[a] Mean diameter is the average of two diameter measurements taken at right angles to each other at any point along the length.

[b] Nominal diameters are those shown in Table III.

TABLE X.—PERMISSIBLE VARIATIONS IN WALL THICKNESS.
(Revision of Table V, Specification B 241)

Pipe Schedule No.	Permissible Variations in Wall Thickness from the Nominal Wall Thickness, per cent or in.[a]
5 and 10............	±12½ per cent, ±0.012 in. min
20 and greater.......	−12½ per cent[b]

[a] Nominal wall thicknesses are those shown in Table III.

[b] Maximum wall thickness is controlled by weight tolerance.

Tentative Specification for Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 – 58 T):

Section 2.—Revise Item (*7*) to read as follows: "(*7*) Whether inspection is required at the manufacturer's works (Section 19(*a*))."

Section 19(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Pipe (B 241 – 58 T):

Section 2.—Revise Item (*8*) to read as follows: "(*8*) Whether inspection is required at the manufacturer's works (Section 16(*a*)), and."

Table I.—For alloy GM51A (5456) delete the beryllium limit and revise the present maximum copper limit of "0.20" to read "0.10" per cent.

Table IV.—Revise to read as shown in the accompanying Table IX.

Table V.—Revise to read as shown in the accompanying Table X.

Section 16(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Die Forgings (B 247 – 58 T):

Section 2.—Revise Item (*5*) to read as follows: "(*5*) Whether inspection is required at the manufacturer's works (Section 16(*a*))."

Section 16(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 – 58 T):

Section 2.—Revise Item (*9*) to read as follows: "(*9*) Whether inspection is required at the manufacturer's works (Section 19(*a*))."

Section 19(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 – 58 T):

Section 2.—Revise Item (*5*) to read as follows: "(*5*) Whether inspection is required at the manufacturer's works (Section 15(*a*))."

Table I.—For alloy GM51A (5456) delete the beryllium limit and revise the present maximum copper limit of "0.20" to read "0.10" per cent.

Section 15(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Round Welded Tubes (B 313 – 58 T):

Section 2.—Revise Item (*7*) to read as follows: "(*7*) Whether inspection is required at the manufacturer's works (Section 17(*a*))."

Section 17(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Rivet and Cold Heading Wire and Rods (B 316 – 58 T):

Section 2.—Revise Items (*7*) and (*8*) to read as follows: "(*7*) Whether marking for identification is required (Section 15), and (*8*) Whether inspection is required at the manufacturer's works (Section 16(*a*))."

Section 16(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Extruded Aluminum-Alloy Bars, Rods, Pipe, and Structural Shapes for Electrical Purposes (Bus Conductors) (B 317 – 58 T):

Section 3.—Revise Item (*6*) to read as follows: "(*6*) Whether inspection is required at the manufacturer's works (Section 19(*a*))."

Section 19(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Tentative Specification for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318 – 58 T):

Section 2.—Revise Item (*5*) to read as follows: "(*5*) Whether inspection is required at the manufacturer's works (Section 17(*a*))."

Section 17(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recomends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specification for Aluminum for Use in Iron and Steel Manufacture (B 37 – 57):

Section 9(a).—Revise to read as indicated above for Section 18(*a*) in Tentative Specification B 209.

Standard Specification for Magnesium, Base Alloy Sand Castings (B 80 – 58):

Section 2.—Revise Item (*7*) to read as follows: "(*7*) Whether inspection is required at the manufacturer's works (Section 15(*a*))."

Table I.—Add alloy K1A with the following chemical requirements: zirconium, 0.40 to 1.0 per cent; other impurities, 0.30 per cent max; magnesium, remainder.

Table II.—Add alloy K1A-F (as cast) with the following tensile requirements: tensile strength, 24,000 psi, min; yield strength, 6000 psi, min; elongation in 2 in., 14 per cent, min.

Table III.—Add alloy K1A-F (as cast) with a yield strength of 6000 psi min and a corresponding unit deformation of 0.0029 in. per in. of gage length.

Section 15(a).—Revise to read as indicated above for Section 14(*a*) in Tentative Specification B 26.

New Explanatory Note.—Add the following under Note 1:

Alloy K1A.—This alloy is a low strength alloy generally used for its exceptionally good damping characteristics.

TABLE XI.—ADDITIONS TO TABLE I, SPECIFICATION B 90.

	Alloy HK31A[c]	Alloy HM21A	Alloy ZE10A
Magnesium, per cent	remainder	remainder	remainder
Aluminum, per cent	...	...	...
Manganese, min, per cent	...	0.45 to 1.1	...
Rare earths, per cent	...	...	0.12 to 0.22
Zinc, per cent	0.30 max	...	1.0 to 1.5
Thorium, per cent	2.5 to 4.0	1.5 to 2.5	...
Zirconium, per cent	0.40 to 1.0	...	...
Silicon, max, per cent	...	...	...
Copper, max, per cent	0.10	...	...
Nickel, max, per cent	0.01	...	...
Iron, max, per cent	...	...	...
Calcium, max, per cent	...	...	...
Other impurities, max, per cent	0.30	0.30	0.30

[c] Analysis for zinc, copper, and nickel need not ordinarily be made, but if determined they shall be within the specified maximum.

Standard Specification for Magnesium-Base Alloy Sheet and Plate (B 90 - 58):

Section 2.—Revise Item (*6*) to read as follows: "(*6*) Whether inspection is required at the manufacturer's works (Section 15(*a*))."

Table I.—Add alloys HM21A and ZE10A and revise chemical requirements for HK31A to read as shown in the accompanying Table XI.

Table II.—Revise to read as shown in the accompanying Table XII.[2]

Section 15(a).—Revise to read as follows:

15. (*a*) If the purchaser desires that inspection be made at the manufacturer's works where the material is made, it shall be so stated in the contract or purchase order.

New Explanatory Note.—Add the following under Note 1:

Alloy HM21A is recommended for high-temperature service, especially in the temperature range of 500 to 700 F.

Alloy ZE10A is a general purpose alloy with good weldability and does not require stress relief after welding.

Table VII.—Revise to read as shown in the accompanying Table XIII.[2]

Standard Specification for Magnesium-Base Alloy Forgings (B 91 - 58):

Section 2.—Revise Item (*7*) to read as follows: "(*7*) Whether inspection is required at the manufacturer's works (Section 14(*a*))."

Section 14(a).—Revise to read as indicated above for Section 15(*a*) in Standard Specification B 90.

Standard Specification for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 93 - 58):

Section 2.—Revise Item (*4*) to read as follows: "(*4*) Whether inspection is required at the manufacturer's works (Section 9(*a*))."

---

[2] The new table is not included in this report but appears in the revised specification, see 1959 Supplement to Book of ASTM Standards, Part 2.

Section 9(a).—Revise to read as indicated above for Section 15(*a*) in Standard Specification B 90.

Standard Specification for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 58):

Section 2.—Revise Item (*7*) to read as follows: "(*7*) Whether inspection is required at the manufacturer's works (Section 15(*a*))."

Section 15(a).—Revise to read as indicated above for Section 15(*a*) in Standard Specification B 90.

TABLE XIV.—ADDITIONS TO TABLE I, SPECIFICATION B 179.

	995A	SC64D
Aluminum, per cent	99.5 min [b]	remainder
Copper, per cent	0.05	3.5 to 4.5
Iron, per cent	0.35 [c]	0.8
Silicon, per cent	0.15 [c]	5.5 to 7.0
Manganese, per cent	0.03	0.50
Magnesium, per cent	...	0.10
Zinc, per cent	...	1.0
Titanium, per cent	...	0.25
Nickel, per cent	...	0.35
Other elements, per cent:		
Each	0.03 [d]	...
Total	0.15	0.50

Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 58):

Section 2.—Delete the present Item (*5*) and replace with the following:

(*5*) Whether electrical resistivity or conductivity tests are required for alloy 995A (Section 9), and

(*6*) Whether inspection is required at the manufacturer's works (Section 15(*a*)).

New Sections.—Add new Sections 9 through 12 to read as follows, renumbering the present Sections 9 through 13 accordingly:

9. *Electrical Resistivity.*—When specified in the contract or purchase order, the electrical resistivity determined on specimens of alloy 995A shall not exceed 0.0831 ohm—gram/meter^2 at 20 C(68 F) corresponding to a volume conductivity of not less than 56 per cent of the International Annealed Copper Standard.

10. *Test Specimens.*—(*a*) Specimens for determining resistivity or conductivity shall be either machined from the ingot or from test bars separately cast in permanent molds, provided that, in case of dispute, the results secured from specimens machined from the ingot shall be the basis for acceptance.

(*b*) The test specimens shall be of suitable size and shape appropriate to the instrument and method of test used in making the determination.

NOTE.—When the eddy current method is used, the determinations shall be made on the ingot.

11. *Number of Tests.*—(*a*) The specimens required for the test prescribed in Section 9 shall be representative of each ingot size in the shipment and selected according to the following schedule:

Number of Ingots in Lot	Minimum Number of Specimens
1 to 50	1
51 to 200	2
201 to 1500	3
Over 1500	0.2 per cent of number of ingots in lot

(*b*) When more than one specimen is required to be taken from the lot, no two specimens shall be taken from the same ingot.

12. *Methods of Testing.*—Electrical resistivity or conductivity shall be determined, insofar as practical, in accordance with Method of Test for Resistivity of Electrical Conductor Materials (ASTM Designation: B 193) or the eddy current method, provided that, in case of dispute, the results secured by Method B 193 shall be the basis for acceptance.

Section 11(a). Renumber as Section 15 and revise to read as indicated above for Section 15(*a*) in Standard Specification B 90.

Table I.—Delete alloys SC64A and SC64B, and add alloys 995A and SC64D with the chemical requirements shown in Table XIV.

Add new Footnotes *b*, *c*, and *d* to read as follows, relettering the present footnotes *b*, *c*, *d*, and *e* accordingly:

[b] By difference. Aluminum content is the difference between 100.00 per cent and the sum

of all other metallic elements present in amounts of 0.10 per cent or more, each, expressed to the second decimal.

[c] Purchaser may specify a minimum iron-to-silicon ratio.

[d] The maximum boron content may be specified by purchaser.

Tables II and III.—Delete alloys SC64A and SC64B and their color codes, and add alloy 995A with the color code "Orange and White" and alloy SC64D with the color code "Green and White."

Standard Specification for Magnesium-Base Alloy Permanent Mold Castings (B 199 – 58):

Section 2.—Revise Item (7) to read as follows: "(7) Whether inspection is required at the manufacturer's works (Section 15(*a*)).

Section 15(*a*).—Revise to read as indicated above for Section 14(*a*) in Tentative Specification B 26.

Standard Specification for Magnesium-Base Alloy Extruded Tubes (B 217 – 58:)

Section 2.—Revise Item (7) to read as follows: "(7) Whether inspection is required at the manufacturer's works (Section 15(*a*))."

Section 15(*a*).—Revise to read as indicated above for Section 15(*a*) in Standard Specification B 90.

Standard Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 – 58):

Section 3(*c*).—Revise to read as follows:

> (*c*) In rounding-off percentages, the nearest whole number shall be used. If two choices are possible as when the decimal is followed by a 5 only, or a 5 followed only by zeros, the nearest even whole number shall be used.

APPENDIX II

CHROMIC ACID ANODIZING CHARACTERISTICS OF WROUGHT ALUMINUM ALLOYS*

By George E. Best,[1] J. G. Hecker, Jr.,[2] John W. McGrew,[3] and R. V. VandenBerg[2]

Synopsis

Experimental preparation of chromic acid anodized coatings has been carried out with the following wrought aluminum alloys: 1100, 2024, 3003, 5052, 5056, 6061, 7075, Alclad 2014, and Alclad 7075. Operating variables of voltage, time, bath pH, and temperature were explored above and below the chosen standard of 40 v, 30 min, pH 0.5, and 95 F. Measurements were made of current density, coating weight, coating thickness, dielectric strength, and salt spray resistance, with calculations of coating weight per amp-min, electrical and coating efficiencies.

Although there has been much discussion in the technical literature of the fundamental character and effective properties of anodic coatings on aluminum (**1–12**),[4] much less has been reported on the relative behavior of different aluminum alloys uuder varying conditions of anodic treatment. This is especially true of chromic acid anodizing (**13–16**) as opposed to other electrolytes (**17–21**). Such information has become of particular interest since the acceptance of anodic coating weight as an alternative to salt spray testing under Military Specification MIL-A-8625A.

In view of the continuing commercial importance of chromic acid anodizing and the belief that more widespread application might result from additional knowledge, especially if it pointed the way toward savings in cost or more intelligent choice of alloys, an investigative program was undertaken. The results of its first phase on selected wrought alloys are reported here, which are intended to serve as the base of departure for further extension of processing variables that offer promise in either improved properties or economies in practice.

Scope of Experiments

Anodizing at 40 v for 30 min in chromic acid solution of pH 0.5 (40 g per liter free CrO_3) at 95 F corresponds to average commercial operation and was accordingly chosen as the standard processing treatment. Bath temperature and pH, time, and voltage—both higher and lower levels—were evaluated as single variables on nine different alloys. Details of alloy composition, anodizing apparatus, panel preparation, and evaluation are given in the Appendix in outline form for easy reference, and test data are tabulated in Tables I through IX.

---

* Presented at the February, 1959, meeting of Committee B-7 at Pittsburgh, Pa.

[1] Solvay Process Division, Allied Chemical Corp., Syracuse, N. Y.

[2] Alcoa Process Development Laboratories, Aluminum Co. of America, New Kensington, Pa.

[3] The Martin Co., Baltimore, Md.; formerly with Solvay Process Division, Allied Chemical Corp.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—TEST DATA FOR ALLOY 1100.

	Current Density, amp per sq ft	Weight, mg per sq ft		Sealed Coating, mg per amp-min	Electrical Efficiency, per cent	Coating Efficiency, per cent	Coating Thickness, mils	Dielectric Strength, v	Salt Spray Rating
		Unsealed	Sealed						
Higher temperature (115 F)	5.6	850	895	5.8	93	51	0.13	320 to 390	4
Longer time (60 min)	2.6	845	980	7.2	98	59	0.21	460 to 600	5
Standard: 40 v, 30 min, 95 F, pH 0.5	3.1	530	590	7.2	96	60	0.09	340 to 400	5
Lower pH (pH 0.2)	2.8	490	550	7.2	95	61	0.09	300 to 350	5
Higher voltage (60 v)	2.9	530	590	7.1	96	61	0.09	270 to 300	5
Higher pH (pH 0.9)	2.6	430	475	6.7	92	58	0.09	210 to 290	4
Lower voltage (20 v)	2.1	330	375	6.6	94	56	0.06	250 to 310	4
Lower temperature (85 F)	1.6	340	370	7.3	99	63	0.06	250 to 320	4
Shorter time (15 min)	3.1	270	300	7.1	95	61	0.06	220 to 280	5

TABLE II.—TEST DATA FOR ALLOY 2024.

	Current Density, amp per sq ft	Weight, mg per sq ft		Sealed Coating, mg per amp-min	Electrical Efficiency, per cent	Coating Efficiency, per cent	Coating Thickness, mils	Dielectric Strength, v	Salt Spray Rating
		Unsealed	Sealed						
Higher temperature (115 F)	5.2	400	495	3.2	70	30	0.08	420 to 520	4
Longer time (60 min)	2.2	345	435	3.4	57	32	0.09	380 to 400	4
Standard: 40 v, 30 min, 95 F, pH 0.5	1.9	170	195	3.4	57	32	<0.04	220 to 300	4
Lower pH (pH 0.2)	2.8	260	285	3.4	63	33	<0.04	340 to 360	3
Higher voltage (60 v)	2.8	230	250	2.8	48	29	<0.04	350 to 400	2
Higher pH (pH 0.9)	1.3	120	140	3.6	56	34	0.05	240 to 280	2
Lower voltage (20 v)	2.4	205	240	3.2	64	31	<0.04	240 to 350	4
Lower temperature (85 F)	1.3	105	120	3.2	51	30	0.05	160 to 240	1
Shorter time (15 min)	2.0	85	95	3.3	54	31	<0.04	160 to 220	1

A discussion of sealing is given here for emphasis. It has been adequately demonstrated in both tests and regular practice that, although chromic acid anodized coatings impart a relatively high degree of basis metal protection without being sealed, the highest level of protection is achieved by sealing the coating, especially when sealing is carried out in a solution containing hexavalent chromium. Presumably this acts as a corrosion inhibitor when absorbed within the anodic coating. In this program, therefore, it was accepted as a basic tenet that anodic coatings applied for protection ought to be sealed, for which 10 min at 200 F in de-ionized water containing just enough chromic acid to adjust to pH 5 constitutes minimum but effective sealing treatment. Lower temperatures may be employed by increasing the time; prolonged boiling is considered undesirable because of possible dissolution of aluminum oxide from the coating.

TABLE III.—TEST DATA FOR ALLOY 3003.

	Current Density, amp per sq ft	Weight, mg per sq ft		Sealed Coating, mg per amp-min	Electrical Efficiency, per cent	Coating Efficiency, per cent	Coating Thickness, mils	Dielectric Strength, v	Salt Spray Rating
		Unsealed	Sealed						
Higher temperature (115 F)	5.0	710	840	5.5	93	53	0.13	270 to 340	4
Longer time (60 min)	2.6	725	860	5.5	88	52	0.17	300 to 350	4
Standard: 40 v, 30 min, 95 F, pH 0.5	2.6	385	450	5.8	89	55	0.10	180 to 240	4
Lower pH (pH 0.2)	2.7	440	465	5.7	94	54	0.10	160 to 250	1M
Higher voltage (60 v)	2.7	430	490	6.1	91	58	0.10	180 to 250	4
Higher pH (pH 0.9)	1.7	260	265	5.3	89	49	0.08	60 to 130	1S
Lower voltage (20 v)	1.6	240	285	5.8	92	55	0.09	100 to 170	1
Lower temperature (85 F)	1.7	270	320	6.2	90	59	0.08	70 to 170	1
Shorter time (15 min)	2.5	200	235	6.2	91	59	0.08	80 to 160	1

TABLE IV.—TEST DATA FOR ALLOY 5052.

	Current Density, amp per sq ft	Weight, mg per sq ft		Sealed Coating, mg per amp-min	Electrical Efficiency, per cent	Coating Efficiency, per cent	Coating Thickness, mils	Dielectric Strength, v	Salt Spray Rating
		Unsealed	Sealed						
Higher temperature (115 F)	6.4	900	890	4.7	91	44	0.16	320 to 420	5
Longer time (60 min)	3.0	900	905	5.0	89	47	0.14	380 to 420	5
Standard: 40 v, 30 min, 95 F, pH 0.5	2.6	405	410	5.5	92	52	0.08	180 to 260	4
Lower pH (pH 0.2)	3.0	460	465	5.2	88	49	0.06	220 to 300	2M
Higher voltage (60 v)	2.7	415	415	5.1	82	48	<0.05	180 to 220	2M
Higher pH (pH 0.9)	2.2	355	360	5.6	92	53	<0.05	100 to 160	5
Lower voltage (20 v)	2.0	300	305	5.0	91	48	<0.05	120 to 200	1M
Lower temperature (85 F)	1.7	285	285	5.5	89	52	<0.05	100 to 200	1M
Shorter time (15 min)	2.6	210	210	5.4	90	51	<0.05	100 to 120	1S

TABLE V.—TEST DATA FOR ALLOY 5056.

	Current Density, amp per sq ft	Weight, mg per sq ft		Sealed Coating, mg per amp-min	Electrical Efficiency, per cent	Coating Efficiency, per cent	Coating Thickness, mils	Dielectric Strength, v	Salt Spray Rating
		Unsealed	Sealed						
Higher temperature (115 F)	8.9	1270	1330	5.0	96	47	0.17	480 to 540	5
Longer time (60 min)	3.4	1130	1200	5.8	102	55	0.11	360 to 420	4S
Standard: 40 v, 30 min, 95 F, pH 0.5	3.4	565	560	5.5	96	52	0.06	260 to 280	4
Lower pH (pH 0.2)	3.3	560	550	5.4	98	52	0.07	240 to 280	3M
Higher voltage (60 v)	2.6	410	420	5.4	90	51	<0.05	180 to 240	5M
Higher pH (pH 0.9)	3.0	495	510	5.7	94	54	0.08	210 to 260	5
Lower voltage (20 v)	2.8	460	450	5.2	97	49	0.06	200 to 250	4M
Lower temperature (85 F)	1.8	305	310	5.6	93	53	<0.05	140 to 180	2M
Shorter time (15 min)	2.7	220	230	5.7	94	54	<0.05	80 to 100	4S

TABLE VI.—TEST DATA FOR ALLOY 6061.

	Current Density, amp per sq ft	Weight, mg per sq ft		Sealed Coating, mg per amp-min	Electrical Efficiency, per cent	Coating Efficiency, per cent	Coating Thickness, mils	Dielectric Strength, v	Salt Spray Rating
		Unsealed	Sealed						
Higher temperature (115 F)	5.0	675	710	4.7	88	44	0.15	370 to 440	5
Longer time (60 min)	2.4	680	720	5.1	87	48	0.13	300 to 360	5
Standard: 40 v, 30 min, 95 F, pH 0.5	2.4	360	375	5.2	88	49	0.07	150 to 290	4
Lower pH (pH 0.2)	2.5	370	390	5.1	87	49	0.08	170 to 220	4
Higher voltage (60 v)	2.1	305	310	5.2	84	49	0.06	150 to 200	1M
Higher pH (pH 0.9)	1.9	275	280	4.8	85	46	0.06	140 to 180	1S
Lower voltage (20 v)	1.9	265	265	4.6	86	44	0.06	110 to 170	1M
Lower temperature (85 F)	1.6	240	245	5.2	86	49	0.07	100 to 150	1S
Shorter time (15 min)	2.4	180	180	5.1	85	48	0.05	80 to 140	1S

TABLE VII.—TEST DATA FOR ALLOY 7075.

	Current Density, amp per sq ft	Weight, mg per sq ft		Sealed Coating, mg per amp-min	Electrical Efficiency, per cent	Coating Efficiency, per cent	Coating Thickness, mils	Dielectric Strength, v	Salt Spray Rating
		Unsealed	Sealed						
Higher temperature (115 F)	3.8	425	435	4.0	87	37	0.08	260 to 320	4
Longer time (60 min)	2.1	465	530	4.9	82	41	0.08	200 to 240	4
Standard: 40 v, 30 min, 95 F, pH 0.5	2.1	255	310	5.9	84	46	0.05	200 to 230	4
Lower pH (pH 0.2)	2.3	265	340	6.0	80	46	0.07	220 to 300	4
Higher voltage (60 v)	1.4	130	150	4.4	65	35	0.06	120 to 170	5
Higher pH (pH 0.9)	2.1	230	275	5.3	78	42	0.06	130 to 150	4
Lower voltage (20 v)	2.7	305	390	6.1	87	45	0.07	270 to 310	5
Lower temperature (85 F)	1.0	170	195	4.1	79	53	<0.04	90 to 100	3
Shorter time (15 min)	2.2	140	155	4.9	83	44	0.06	100 to 150	3

TABLE VIII.—TEST DATA FOR ALCLAD 2014.

	Current Density, amp per sq ft	Weight, mg per sq ft		Sealed Coating, mg per amp-min	Electrical Efficiency, per cent	Coating Efficiency, per cent	Coating Thickness, mils	Dielectric Strength, v	Salt Spray Rating
		Unsealed	Sealed						
Higher temperature (115 F)	5.1	720	850	5.6	91	53	0.14	280 to 370	4
Longer time (60 min)	2.5	725	870	5.8	91	55	0.17	300 to 360	4
Standard: 40 v, 30 min, 95 F, pH 0.5	2.4	385	445	6.2	93	58	0.10	100 to 230	4
Lower pH (pH 0.2)	2.2	365	380	5.7	92	54	0.07	100 to 280	1
Higher voltage (60 v)	2.2	370	415	6.3	93	60	0.08	70 to 150	2
Higher pH (pH 0.9)	1.8	285	295	5.4	92	51	0.08	40 to 70	1M
Lower voltage (20 v)	1.7	255	305	5.9	95	56	0.07	80 to 190	1
Lower temperature (85 F)	1.5	260	300	6.6	94	63	0.06	160 to 200	1
Shorter time (15 min)	2.4	200	240	6.8	98	65	0.06	100 to 110	1

TABLE IX.—TEST DATA FOR ALCLAD 7075.

	Current Density, amp per sq ft	Weight, mg per sq ft		Sealed Coating, mg per amp-min	Electrical Efficiency, per cent	Coating Efficiency, per cent	Coating Thickness, mils	Dielectric Strength, v	Salt Spray Rating
		Unsealed	Sealed						
Higher temperature (115 F)	5.2	790	910	5.9	97	56	0.15	390 to 420	4
Longer time (60 min)	2.7	855	965	6.0	96	57	0.17	350 to 460	5
Standard: 40 v, 30 min, 95 F, pH 0.5	2.7	440	490	6.1	94	58	0.10	260 to 340	4
Lower pH (pH 0.2)	2.5	435	455	6.2	96	57	0.10	200 to 310	2
Higher voltage (60 v)	2.6	470	520	6.8	100	64	0.10	230 to 330	3
Higher pH (pH 0.9)	1.8	310	320	5.8	96	55	0.08	60 to 160	1M
Lower voltage (20 v)	1.7	255	300	6.1	97	58	0.07	190 to 240	1
Lower temperature (85 F)	1.7	300	340	6.9	98	65	0.08	160 to 240	1
Shorter time (15 min)	2.6	225	260	6.7	98	63	0.07	100 to 180	2

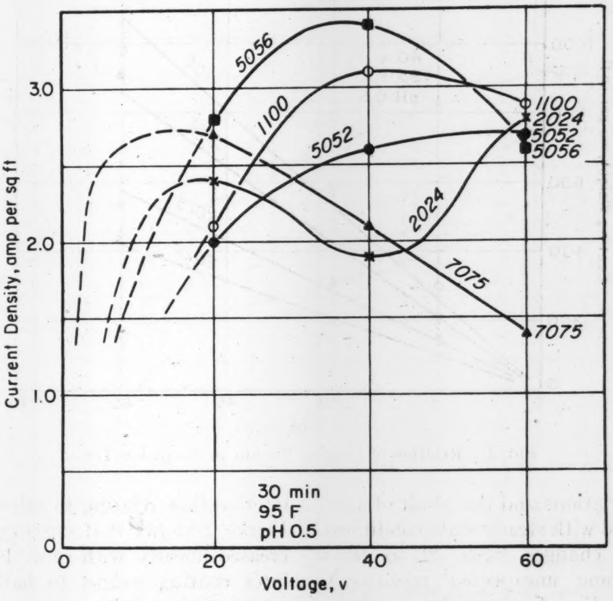


FIG. 1.—Effect of Voltage on Current Density.

DISCUSSION OF RESULTS

Current Density:

Temperature and pH of the anodizing bath had the anticipated influences on current density, in that rising temperature and lowering pH—both improving electrolyte conductivity and dissolving action at the base of the pore cells of the coating—tended to increase current density. However, pH change over the range of 0.9 to 0.2 had generally less effect in degree than might have been supposed. On alloy 2024 the current doubled, and increased 50 per cent on alloy 5052; alloys 3003 and Alclad 7075 showed about 50 per cent increase between pH 0.9 and 0.5, and little change from pH 0.5 to 0.2. Lesser increases were observed with the other alloys.

Current density increased progressively with rising temperature over the range of 85 to 115 F. Roughly, current increased 50 per cent from 85 to 95 F, and doubled from 95 to 115 F. Although observed current densities for each alloy at different anodizing times were not identical, the differences could be experimental variations and the result of averaging initial with steady state conditions.

Voltage changes from 20 to 60 v yielded some unexpected results. As plotted in Fig. 1, these data make it clear that current density passes through a maximum as a function of voltage, and that maximum current density occurs at different voltage levels for the various alloys. It is apparent that the peak for alloy 7075 is reached at a comparatively low voltage—probably below 20 v—thus explaining the common problem of low anodic coating weight on this alloy when processed at the conventional 40 v. Alloys 1100, 5056, 6061, Alclad 2014, and Alclad 7075 peaked within the 20- to 60-v range, indicating 40 v to be near the optimum. Current continued to rise up to 60 v with alloys 3003 and 5052. Alloy 2024 showed a double inflection with a dip at 40 v.

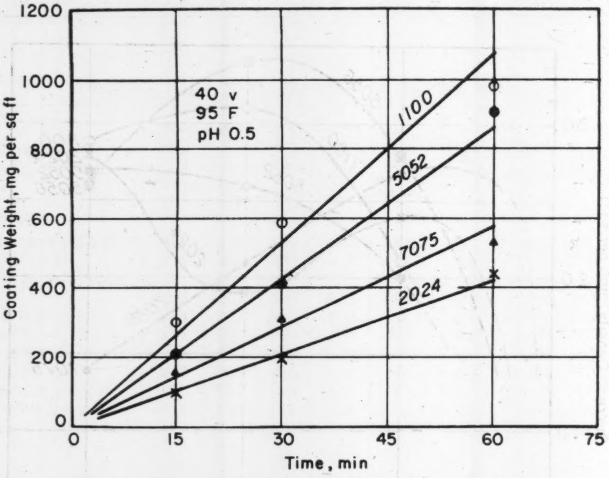


FIG. 2.—Relation of Coating Weight to Anodizing Time.

Coating Weight:

Detailed discussion of coating weights and their significance will be given in a later section relating to salt spray tests. Figure 2 shows that coating weight increased linearly with time. Figure 3 relates coating weight to bath temperature; coating weights were consistently about 50 per cent greater at 95 than at 85 F, with some divergence from doubling in weight from 95 to 115 F.

Coating weight variations followed the same pattern as current density, indicating current density to be highly useful for operating control purposes—a point little recognized or utilized in commercial practice. Utility of current density for operating practice may be further enhanced by incorporating the time factor, thus obtaining an over-all index of sealed coating weight in mg per amp-min. These values spread from about 7 with alloy 1100 to 3 with alloy 2024, all others being intermediate. Higher temperature (115 F) in most instances resulted in a slight to moderate drop in efficiency of coating production.

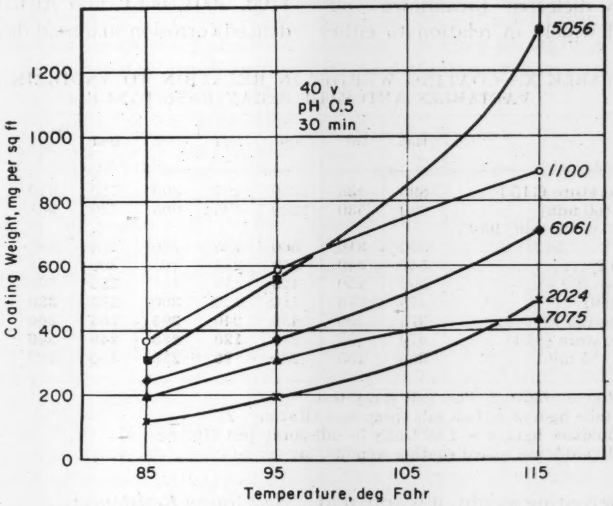


FIG. 3.—Relation of Coating Weight to Bath Temperature.

Electrical Efficiency:

The efficiency of converting aluminum to aluminum oxide was 90 per cent or higher, except for alloys 2024, 6061, and 7075, where efficiency suffers from electrolytic dissolution of copper, zinc, and magnesium constituents, this effect being most pronounced at higher voltage.

Coating Efficiency:

Rule-of-thumb in chromic acid anodizing practice considers that about half of the aluminum oxide formed is retained in the coating, the remainder being dissolved. Most of the dissolved alumina comes from the base of the cell pores, its dissolution being necessary to the formation of coatings of substantial thickness (8).

These data confirm that for most of the alloys tested, coating efficiency is of the order of 50 per cent, without any definite pattern of variation relating to anodizing conditions. Alloys 2024, 6061, and 7075 showed lower efficiency, alloy 2024 being the lowest at about 30 per cent.

Coating Thickness:

While moderate differences in coating density were indicated from varying relationships between thickness and coating weight, thickness broadly followed the same pattern as weight. Accordingly, as with coating weight, thickness essentially doubled with time increase from 30 to 60 min or temperature rise from 95 to 115 F. Thickness change in going from 15 to 30 min or from 85 to 95 F was generally less pronounced and less consistent.

Dielectric Strength:

Dielectric strength of chromic acid anodized coatings is of particular interest in certain applications because of superior resistance, as compared to sulfuric acid coatings, to fracture on bending (22) and to crazing on heating. Thus a comparison of these data with one another is less significant than the individual values for the specific alloys.

Although dielectric breakdown voltages varied widely in relation to either thickness or coating weight, it is apparent that the two conditions tending to produce relatively high coating weight and thickness also yielded the highest dielectric strength measurements.

Outdoor Exposure:

As an adjunct to laboratory examination, single panels were subjected to outdoor exposure, facing south at 45 deg inclination at New Kensington, Pa. Exposures differed from 8 months for alloy 2024 to 34 months for alloys 1100 and 7075, and the others either 14 or 17 months.

There was no significant pitting on the face side of any panel. Incipient attack on Alclad 2014 was discernible on all but the heavier coatings. The lowest weight coatings on alloys 1100 and 2024, and all but the heaviest coating on alloy 7075 largely disappeared by weathering away; that is, the bright metal base was exposed when the panels were cleaned with water and a mild wax base polish.

The back side (under face) of the alloy 7075 and Alclad 2014 panels were appreciably attacked, while those of alloys 1100, 3003, 6061, and Alclad 7075 evidenced corrosion to a mild degree.

TABLE X.—COATING WEIGHT IN RELATION TO ANODIZING VARIABLES AND SALT SPRAY RESISTANCE.

Alloys	1100	7075	5056	2024	5052	6061	Alclad 7075	3003	Alclad 2014
Higher temperature (115 F)	895	435	1330	495	890	710	910	840	850
Longer time (60 min)	980	530	1200	435	905	720	965	860	870
Standard: 40 v, 30 min, 95 F, pH 0.5	590	310	560	195	410	375	490	450	445
Lower pH (0.2)	550	340	550	285	*465*	390	*455*	**465**	**380**
Higher voltage (60 v)	590	150	420	*250*	*415*	**310**	520	490	*415*
Higher pH (0.9)	475	275	510	*140*	360	**280**	**320**	**265**	**295**
Lower voltage (20 v)	375	390	450	240	**305**	**265**	**300**	**285**	**305**
Lower temperature (85 F)	370	195	*310*	**120**	**285**	**245**	**340**	**320**	**300**
Shorter time (15 min)	300	155	230	**95**	**210**	**180**	*260*	**235**	**240**

Legend: Roman digits = Pass salt spray test.
Italic figures = Fail salt spray test (Rating—2).
Boldface figures = Fail badly in salt spray test (Rating—1).
Tabulated values are sealed coating weights, mg per sq ft.

Salt Spray Resistance:

Table X summarizes individual alloy salt spray results for comparison between alloys with respect to anodic coating weights. Three points are clearly evident:

1. While a minimum sealed coating weight of 200 mg per sq ft denoted passable protection for alloy 2024—the higher voltage coating being an exception—it was not indicative of similarly good protection with other alloys.

Alloys 1100, 7075, and 5056 appeared naturally resistant to salt spray attack, and coating weight or anodizing conditions were not critical. This demonstrates that concern about attaining 200 mg per sq ft on alloy 7075 is unwarranted.

At the other end of the scale, Alclad 2014 was the most susceptible of the alloys tested, thus correlating with the rough indication from outdoor exposure.

2. For each alloy there was a coating weight above which satisfactory salt spray protection was afforded. This generality is apparent from inspection of the tabulated values.

3. Anodizing to produce at least as much coating as obtained at the arbitrarily selected standard conditions, that is, 30 min at 40 v in anodizing bath at pH 0.5 and 95 F, yielded satisfactorily protective coatings even though this combination of operating variables was not optimum for all alloys for coating production. This suggests the use of alloy 2024 as a general reference standard for wrought alloys for purpose of coating weight determination, and the establishment of acceptability on the basis of coating weight as a criterion of operating practice.

Higher anodizing bath temperature and longer time have been extensively employed in the production of very heavy coatings to be colored, and higher temperature alone has been explored for the preparation of opaque coatings (23). It is understandable that longer time would have little appeal in the protective coating field since it would add to cost. It is obvious from the data here presented, however, that increasing the temperature might permit shortening the time. This would result in many economic advantages, especially in automated operations. Since either increasing temperature from 95 to 115 F or increasing time from 30 to 60 min approximates doubling the coating weight, one might speculate that 15 to 20 min at 115 F could produce satisfactorily protective coatings. In a separate isolated test a sealed coating of 185 mg per sq ft on alloy 2024 produced in 15 min at 40 v, in a bath at a pH of 0.6 at 110 F, with a current density of 4.1 amp per sq ft, was unaffected after 2800 hr of salt spray test exposure. As this program of work continues, one of its most important phases will be the investigation of short time - higher temperature combinations.

A further example of wide departure from the norm was a coating, also on alloy 2024, prepared in 25 per cent chromic acid, anodized at only 10 v for 15 min at 110 F, with a current density of 9 amp per sq ft, resulting in a coating weight of 360 mg per sq ft. After sealing, this was similarly resistant to 2800 hr of salt spray testing.

Coating Appearance:

It has been commonly observed that chromic acid anodizing of alloy 2024 under the usual conditions similar to those chosen as standard in this program results in a dull, moderately dark grey coating. Likewise, darkening of alloy 7075 was observed. With the other alloys tested there was little change in appearance compared to the basis metal.

The effect of higher voltage was striking in its effect on the appearance of the coatings on alloys 2024 and 7075 in that darkening was eliminated. With the other alloys, there was no noticeable change brought about by voltage increase.

Since this appears to be a specific effect of voltage, it may prove useful in instances where anodic coatings are to be used as the final finishing treatment and where darkening is harmful to appearance. Furthermore, it may open the way for decorative application of chromic acid anodized coatings, perhaps in conjunction with elevated anodizing bath temperatures to produce much higher coating weights.

SUMMARY AND PRINCIPAL CONCLUSIONS

Experimental preparation of chromic acid anodized coatings has been carried out with the following wrought aluminum alloys: 1100, 3003, 2024, 5052, 5056, 6061, 7075, Alclad 2014, and Alclad 7075. Operating variables of voltage, time, bath pH, and temperature were explored above and below the chosen standard of 40 v, 30 min, pH 0.5, and 95 F. Measurements were made of current density, coating weight, coating thickness, dielectric strength, and salt spray resistance, with calculations of coating weight per amp-min, electrical and coating efficiencies.

Current density, in combination with time, was found to bear a direct relation to coating weight for each alloy and is suggested as being a useful control for operating practice. Current density roughly doubled for all alloys from 95 to 115 F. Although increasing acidity (decreasing pH) increased current density, this effect was moderate in degree.

Voltage variations revealed the fact that current density passes through a maximum, with the possibility of more than one inflection, occurring at a different voltage level for each alloy.

There was a level of coating weight above which satisfactory salt spray resistance was obtained, but this was different for each alloy. The amount of coating produced by the chosen standard conditions sufficed. Thus 200 mg per sq ft was not found properly applicable to all alloys as a criterion of adequate corrosion resistance, leading to the suggestion that alloy 2024, for which this minimum coating weight is applicable, be used as a reference standard for operating practice.

The advantage of higher temperature in obtaining more coating weight has been substantiated, with a preliminary indication that reduction in anodizing time—with attendant saving in cost—may be possible.

A specific effect of voltage on coating appearance was observed, notably elimination of darkening in the case of alloys 2024 and 7075 by anodizing at 60 v.

APPENDIX

DETAILS OF EXPERIMENTS

A. *Alloy Selection (Nominal Compositions):*

1100—99.0 aluminum (minimum).
2024—4.5 Cu, 1.5 Mg, 0.6 Mn.
3003—1.2 Mn.
5052—2.5 Mg, 0.25 Cr.
5056—5.2 Mg, 0.1 Cr, 0.1 Mn.
6061—1.0 Mg, 0.25 Cr, 0.6 Si, 0.25 Cu.
7075—5.6 Zn, 2.5 Mg, 1.6 Cu, 0.3 Cr.
Alclad 2014:
  Cladding (6003)—0.7 Si, 0.8 Mn, 1.0 Mg.
  Base (2014)—4.4 Cu, 0.8 Si, 0.8 Mn, 0.4 Mg.
Alclad 7075:
  Cladding (7072)—1.0 Zn, 0.1 Mg, 0.1 Cu, 0.1 Mn.
  Base (7075)—5.6 Zn, 2.5 Mg, 1.6 Cu, 0.3 Cr.

B. *Equipment:*

1. *Anodizing Tank.*—Cylindrical; 9-gal capacity; plain steel, serving also as cathode; thermostatically regulated within 0.5 F range of desired temperature level; mechanical stirrer.
2. *Rack.*—Insulated alloy 2024, accommodating ten 3- by 6-in. panels spaced 0.5 in. apart. (Satisfactory coating uniformity established by direct test.)
3. *Electrical Source.*—Selenium rectifier of 75 v, 100 amp direct current capacity. Voltage raised from 0 to desired level in 2 to 4 min, this being included as part of the anodizing time.
4. *Electrical Measurement.*—A direct-current w-h meter modified by applying a constant voltage so that disk revolu-

tions measured current only; calibrated with a standard copper coulometer.

C. *Chromic Acid:*

Technical grade: 99.75 per cent CrO_3 minimum, 0.1 per cent SO_4 maximum. Anodizing baths renewed frequently to avoid accumulation of impurities or trivalent chromium.

Bath pH	Free CrO_3 Concentration, g per liter
0.9	17
0.5	40
0.2	80

Bath pH is a direct and significant measurement of free chromic acid concentration which determines bath activity so far as anodizing is concerned. Total chromic acid concentration, including the amount neutralized by dissolved alumina, etc. as well as the free chromic acid, may be considerably higher—in commercial practice ranges up to 100 g per liter ("10 per cent CrO_3") or more (15).

D. *Panel Preparation:*

1. Clean with acetone.
2. Three-minute dip in Alcoa R-3 etch (nominal composition: 35 g per liter CrO_3, 175 g per liter H_2SO_4) at 180 F.
3. Wash and dry.

E. *Post-Anodizing Treatment—Sealing:*

Ten minutes at 200 F in de-ionized water adjusted to pH 5 with chromic acid.

F. *Anodized Panel Testing:*

1. Coating weight: ASTM Method B 137. Strip to constant weight in boiling solution containing 20 g per liter CrO_3 and 35 ml 85 per cent H_3PO_4 per liter.
2. Coating thickness: microscopically.
3. Dielectric strength: ASTM Method B 110, employing alternating current.
4. Salt spray resistance: ASTM Method B 117, using 20 per cent NaCl solution.

Duration of test: 250 hr.

Arbitrary rating method as follows:

5—No corrosion.
4—1 to 5 corrosion spots per panel.
3—6 to 10 corrosion spots per panel.
2—11 to 25 corrosion spots per panel.
1—More than 25 corrosion spots per panel.
M—Moderate staining.
S—Severe staining.

Thus ratings of 5 and 4 denote clearly satisfactory resistance; 3 to be borderline; 2 and 1 failure.

G. *Calculations:*

1. Current density: average value for entire anodizing period, calculated from total amount of electricity drawn and anodizing interval.
2. Electrical efficiency, per cent =

$$\frac{\text{mg Al}^{a} \times 1074}{\text{coulombs}}$$

---

[a] Converted to Al_2O_3, being the difference between original weight and weight after stripping anodic coating.

Coating efficiency, per cent =

$$\frac{\text{mg unsealed coating weight} \times 568}{\text{coulombs}}$$

REFERENCES

(1) F. Keller, G. W. Wilcox, M. Tosterud, and C. J. Slunder, "Anodic Coating of Aluminum—Behavior of Alloy Constituents," *Metals and Alloys*, Vol. 10, No. 7, pp. 13–19, July, 1939.

(2) J. D. Edwards, "Thickness of Anodic Coatings on Aluminum," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 959–965 (1940).

(3) H. G. Arlt, "The Abrasion Resistance of Anodically Oxidized Coatings on Aluminum," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 967–975 (1940).

(4) K. G. Compton and A. Mendizza, "Electrical Breakdown of Anodically Oxidized Coatings on Aluminum," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 978–987 (1940).

(5) R. B. Mason and W. C. Cochran, "Meas-

urement of Thickness of Oxide Coatings on Aluminum Alloys," ASTM BULLETIN, No. 148, Oct., 1947, pp. 47–51.

(6) R. L. Burwell, Jr., W. C. Sierichs, and W. E. McConnaughey, "Comparison of the Corrosion Resistance of Aluminum Alloy Assemblies Anodized in Sulfuric and in Chromic Acid," Naval Research Laboratory Report P-2656, Oct., 1945; Publication Board Report PB 109463.

(7) R. C. Renshaw, L. Wallerstein, Jr., and S. J. Zand, "Fatigue Life of Aircraft Engine Mounting Components," SAE Annual Meeting Reprint 405, Jan., 1950.

(8) F. Keller, M. S. Hunter, and D. L. Robinson, "Structural Features of Oxide Coatings on Aluminum," *Journal*, Electrochemical Soc., Vol. 100, No. 9, pp. 411–419, Sept., 1953.

(9) M. S. Hunter and P. E. Fowle, "Determination of Barrier Layer Thickness of Anodic Oxide Coatings," *Journal*, Electrochemical Soc., Vol. 101, No. 9, pp. 481–485, Sept., 1954.

(10) M. S. Hunter and P. E. Fowle, "Factors Affecting the Formation of Anodic Oxide Coatings," *Journal*, Electrochemical Soc., Vol. 101, No. 10, pp. 514–519, Oct., 1954.

(11) R. V. VandenBerg, "What You Should Know About Anodized Coatings for Aluminum," *Materials and Methods*, Vol. 44, No. 1, pp. 90–94, July, 1956.

(12) R. L. Saur, "Nondestructive Thickness Measurement of Anodizing Using the Interference Microscope," *Plating*, Vol. 44, No. 10, pp. 1079–1082, Oct., 1957.

(13) G. D. Bengough and H. Sutton, "The Protection of Aluminum and Its Alloys Against Corrosion by Anodic Oxidation," *Engineering*, Vol. 122, p. 274, Aug. 27, 1926.

(14) R. W. Buzzard, "Anodizing of Aluminum Alloys in Chromic Acid Solutions of Different Concentrations," National Bureau of Standards Research Paper RP 975 (1937).

(15) O. F. Tarr, Marc Darrin, and L. G. Tubbs, "Anodic Treatment of Aluminum in the Chromic Acid Bath," *Industrial and Engineering Chemistry*, Vol. 33, No. 12, pp. 1575–1580, Dec., 1941.

(16) G. E. Best, "Chromic Acid Anodizing of Aluminum," Chapter 15C, "Electroplating Engineering Handbook," Reinhold Publishing Co., New York, N. Y. (1955).

(17) R. B. Mason and C. J. Slunder, "Anodic Reactions of Aluminum and Its Alloys in Sulfuric and Oxalic Acid Electrolytes," *Industrial and Engineering Chemistry*, Vol. 39, No. 12, pp. 1602–1607, Dec., 1947.

(18) R. B. Mason and P. E. Fowle, "Anodic Behavior of Aluminum and Its Alloys in Sulfuric Acid Electrolytes," *Journal*, Electrochemical Soc., Vol. 101, No. 2, pp. 55–59, Feb., 1954.

(19) R. B. Mason, "Factors Affecting the Formation of Anodic Oxide Coatings in Sulfuric Acid Electrolytes," *Journal*, Electrochemical Soc., Vol. 102, No. 12, pp. 671–675, Dec., 1955.

(20) R. C. Spooner, "The Sealing of Sulfuric Acid Anodic Films on Aluminum," *Proceedings*, 44th Annual Meeting, Am. Electroplaters Soc., pp. 132–142 (1957).

(21) R. F. Hafer, "New Developments in Anodizing Aluminum," *Plating*, Vol. 45, No. 6, pp. 623–628, June, 1958.

(22) U. S. Patent 2,788,317, Carlo Sonnino, April 9, 1957.

(23) A. W. Brace, "Production and Properties of Opaque Coatings by Chromic Acid Anodizing," *Transactions*, Inst. Metal Finishing, Vol. 34, Advance Copy No. 3 (1957).

APPENDIX III

ATMOSPHERIC EXPOSURE OF WROUGHT ALUMINUM AND MAGNESIUM ALLOYS*

By L. H. Adam and Marie Dougherty[1]

During the past few years, Committee B-7 on Light Metals and Alloys, Cast and Wrought, has been reporting data relative to the atmospheric effects on aluminum and magnesium alloys exposed at five ASTM test sites throughout the country. The program includes sand and permanent mold castings as well as wrought material tested after exposure periods of six months, one, three, five, and ten years. Information recorded on the wrought samples after 6-months and 1-yr exposure had been reported to the Society in 1955,[2] while data on the castings were summarized in the 1954[3] and 1958[4] ASTM *Proceedings*. Therefore, this particular report will pertain only to the wrought aluminum and magnesium alloys exposed for 3 yr at the rural test site, State College, Pa., the industrial site, New York, N. Y., and the marine sites at Kure Beach, N. C., Point Reyes, Calif., and Freeport, Tex.

Evaluations for the sheets (0.064 in.), the plates and extruded bars (0.25 in.) are based on the average tensile strength and per cent elongation obtained by testing four standard specimens[5] machined from each alloy after exposure at the various test sites. Values for the aluminum riveted samples represent the average of six specimens per alloy, while the magnesium riveted values are based on average results of three specimens. Tensile strength was determined on all specimens with the exception of the spot welded material. In this instance, only breaking loads were recorded. Values are compared with tests made on a similar number of samples retained in sealed containers under control conditions for a three year period. Tables I through XXXIV contain the actual mechanical properties obtained on the various alloys with their corresponding ASTM Specifications. Figures 1 through 5 are bar graphs showing the average for each alloy at the five test sites as compared with the average control values for the same period.

It is noted on both aluminum and

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Materials Engineer and Physical Science Aide, respectively, Frankford Arsenal, Pitman Dunn Laboratories, Philadelphia, Pa.

[2] L. H. Adam and M. Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," Appendix to Report of Committee B-7 on Light Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 55, pp. 248–311 (1955).

[3] L. H. Adam, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," Appendix to Report of Committee B-7 on Light Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 270 (1954).

[4] L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," Appendix to Report of Committee B-7 on Light Alloys Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 194 (1958). See p. 27.

[5] Tentative Method of Tension Testing of Metallic Materials Designation: E 8 – 57 T, 1958 Book of ASTM Standards, Part 3.

magnesium alloys 0.064 and 0.25 in. thick, that the general decrease in tensile strength after 3-yr exposure is less than 5 per cent. The percentage elongation, however, chiefly on the magnesium alloys, shows a marked decrease when compared with the control values. Less than a 20 per cent average decrease in ductility is recorded on aluminum alloys, while approximately a 40 per cent average decrease is shown in the magnesium samples. In considering the aluminum alloys, it appears that the unclad aluminum-copper samples show the greatest decrease in strength and ductility after exposure. This fact is particularly noticeable at the Point Reyes, Calif. test site. Apparently, there is very little change in the physical properties of clad material after a 3-yr exposure. With reference to the magnesium alloys, the M1A code shows the greatest decrease in strength and ductility. There is no significant change noted in the welded and riveted samples.

Acknowledgment is extended to the various participating members of the Society whose excellent cooperation has made such an extensive program possible. Particular mention at this time is given to Reynolds Metals Co. and Aluminum Company of America for testing the wrought aluminum alloys, and to the Bell Telephone Laboratories for being responsible for tests on the magnesium alloy samples. As these data are reported upon expiration of the various exposure periods, it should prove beneficial to those interested in evaluating the performance of such light metals.

TABLE I. - DATA ON ALUMINUM ALLOY NO. M1A-H14 (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements[a]	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
No Requirements	21300	21500	21700	22400	21900	21500
	21500	21600	21000	21700	21400	21200
	21500	21300	21100	21700	21700	20400
	22000	21300	20800	21700	21900	20500
Average.	21600	21400	21200	21900	21700	20900
TENSILE STRENGTH, PSI						
20000 psi, min	23000	22800	22200	23100	22700	22400
	23000	22600	21900	22500	22400	22300
	23200	22600	22000	22500	22400	21900
	23100	22700	21900	22700	22500	21600
Average.	23100	22700	22000	22700	22500	22000
ELONGATION IN 2 IN., PER CENT						
5.0 per cent, min	9.0	7.5	6.5	8.0	8.0	6.0
	9.0	8.5	7.0	8.0	7.0	6.0
	9.0	8.0	6.0	8.0	7.0	6.0
	9.0	8.0	6.5	9.0	7.0	6.0
Average.	9.0	8.0	6.5	8.2	7.2	6.0

[a] Tentative specifications for aluminum - alloy, sheet and plate (B 209-56T)

TABLE II. - DATA ON ALUMINUM ALLOY NO. GR20A-H34 (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
No Requirements	28500	28400	26700	28000	28600	27000
	27200	27100	26700	28200	28100	26800
	28000	28400	26800	28000	27600	27000
	27900	28300	26500	28000	27800	26800
Average.	27900	28000	26700	28000	28000	26900
TENSILE STRENGTH, PSI						
34000 psi, min	35200	34400	33800[a]	35200	34800	32800[a]
	36000	35600	33300[a]	35000	34800	33300[a]
	36000	35300	33500[a]	34800	34900	33000[a]
	35300	35600	33500[a]	35000	34900	32900[a]
Average.	35600	35200	33500[a]	35000	34800	33000[a]
ELONGATION IN 2 IN., PER CENT						
6.0 per cent, min	11.5	10.0	9.5	10.5	10.0	9.0
	10.0	10.0	9.5	10.0	11.0	10.0
	10.5	10.0	10.5	10.5	11.0	10.0
	10.5	10.0	10.5	10.0	11.0	10.0
Average.	10.6	10.0	9.9	10.2	10.8	9.8

[a] LOW

TABLE III. - DATA ON ALUMINUM ALLOY NO. GS11A-T6 (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
35000 psi, min	43500	39400	39700	41800	40500	42500
	42700	39200	39400	41900	40000	41900
	42700	37200	39200	42000	38900	41800
	42700	39200	39900	43000	38700	41800
Average.	42900	38800	39600	42200	39500	42000
TENSILE STRENGTH, PSI						
42000 psi, min	47800	46800	44600	47300	46400	45400
	47500	46900	44100	47000	45800	45300
	47400	45700	44100	47100	44700	45700
	47200	47200	44500	47500	44900	45500
Average.	47500	46600	44300	47200	45400	45500
ELONGATION IN 2 IN., PER CENT						
10.0 per cent, min	10.0	14.0	11.5	12.5	11.0	10.0
	10.0	13.0	12.5	12.0	11.0	10.0
	10.0	13.5	12.0	13.0	11.0	10.0
	11.0	13.0	12.8	10.5	11.0	10.0
Average.	10.2	13.4	12.2	12.0	11.0	10.0

TABLE IV. - DATA ON ALUMINUM ALLOY NO. CG42A-T3 (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
42000 psi, min	48700	45600	45800	47200	b 45400	45000
	48400	48000	46000	48500	44400	44000
	47700	47900	44900	48400	43600	45400
	47600	47100	46900	47200	44400	42100
Average.	48100	47200	45900	47800	44400	44100
TENSILE STRENGTH, PSI						
64000 psi, min	69600	70700	65200	66000	59500^{a}	63500^{a}
	69400	71200	64600	66200	49700^{a}	62800^{a}
	69200	70900	63800^{a}	67200	56300^{a}	64300
	69700	70300	64000	66500	59100^{a}	63000^{a}
Average.	69500	70800	64400	66500	56200^{a}	63400^{a}
ELONGATION IN 2 IN., PER CENT						
17.0 per cent, min	18.0	19.0	15.0^{a}	13.0^{a}	5.2^{a}	13.0^{a}
	18.5	19.0	15.0^{a}	14.0^{a}	2.0^{a}	14.0^{a}
	18.0	18.0	14.5^{a}	14.0^{a}	5.0^{a}	14.0^{a}
	19.0	18.5	14.5^{a}	14.0^{a}	7.0^{a}	14.0^{a}
Average.	18.4	18.6	14.8^{a}	13.8^{a}	4.8^{a}	13.8^{a}

[a] LOW

[b] Samples badly corroded

TABLE V. - DATA ON ALUMINUM ALLOY NO. ZG62A-T6 (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
66000 psi, min	70100 71100 71200 71400	68100 67700 68500 68300	72100 69900 69800 71200	73600 71400 69900 72900	69500 68400 68700 69300	71100 71200 72200 72400
Average.	71000	68200	70800	72000	69000	71700
	TENSILE STRENGTH, PSI					
77000 psi, min	81300 81600 82000 82100	79400 79200 79700 79400	78300 77400 77100 78100	80300 80300 79800 80600	78200 78400 78500 78300	77300 77600 77600 77900
Average.	81800	79400	77700	80200	78400	77600
	ELONGATION IN 2 IN., PER CENT					
8.0 per cent, min	10.0 10.0 10.0 10.0	11.0 10.0 10.0 10.0	9.0 9.0 9.0 9.0	9.5 9.5 9.5 9.0	7.0[a] 9.0 9.0 8.0	9.0 9.0 9.0 9.0
Average.	10.0	10.2	9.0	9.4	8.2	9.0

[a] LOW

TABLE VI. - DATA ON ALUMINUM ALLOY NO. MG11A-H34 (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
No Requirement	31200 31600 31900 32000	32100 29800 31800 30900	29400 29100 29300 29200	32100 32000 32100 32100	30800 29900 30500 30000	29500 29100 28900 29000
Average.	31700	31200	29200	32100	30300	29100
	TENSILE STRENGTH, PSI					
32000 psi, min	36800 36700 36700 37500	37100 36900 37400 36600	35600 35000 34800 35100	36000 36000 36300 36200	36300 36300 36300 36700	35400 35400 35800 35300
Average.	36900	37000	35100	36100	36400	35500
	ELONGATION IN 2 IN., PER CENT					
4.0 per cent, min	9.0 9.0 9.0 9.0	9.0 9.0 9.0 9.0	8.0 8.0 8.0 7.3	8.0 8.0 8.0 8.0	8.0 8.0 8.0 10.0	8.0 8.0 8.0 8.0
Average.	9.0	9.0	7.8	8.0	8.5	8.0

TABLE VII. - DATA ON ALUMINUM ALLOY NO. G1A-H34 (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
No Requirement	24000 23800 23000 23700	23700 23700 23700 24300	22900 22800 22800 22500	24200 24300 24200 23900	24200 24300 24100 24600	23300 23200 23200 23000
Average.	23600	23800	22800	24200	24300	23200
		TENSILE STRENGTH, PSI				
25000 psi, min	28400 28300 27100 27700	27800 27500 27500 27700	26500 26500 26500 26500	27600 27600 27400 27600	27700 27500 27300 27900	26700 26600 26700 26700
Average.	27900	27600	26500	27600	27600	26700
		ELONGATION IN 2 IN., PER CENT				
5.0 per cent, min	9.0 9.0 8.0 8.0	8.5 8.5 8.5 8.5	8.0 8.0 7.0 7.0	8.5 8.5 8.5 8.0	9.0 8.0 7.0 7.0	6.0 6.0 6.0 6.0
Average.	8.5	8.5	7.5	8.4	7.8	6.0

TABLE VIII. - DATA ON ALUMINUM ALLOY NO. CS41A-T6 (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
57000 psi, min	59500 60600 60400 58700	59400 59100 57500 59200	60200 60700 60500 60400	59800 58800 60200 59600	— 58600 59100 59000	58100 59600 58600 59100
Average.	59800	58800	60400	59600	58900	58800
		TENSILE STRENGTH, PSI				
64000 psi, min	67500 67900 67800 65500	67300 67400 65100 67100	66100 66000 66000 66000	66700 66100 66100 66300	66500 65700 66700 66500	64500 65300 65100 64600
Average.	67200	66700	66000	66300	66400	64900
		ELONGATION IN 2 IN., PER CENT				
8.0 per cent, min	9.0 9.0 9.0 9.0	9.5 9.5 9.5 9.5	9.0 9.0 8.5 9.0	9.0 9.0 9.0 9.0	9.0 10.0 9.0 10.0	9.0 9.0 8.0 8.0
Average.	9.0	9.5	8.9	9.0	9.5	8.5

TABLE IX. - DATA ON ALUMINUM ALLOY NO. CG42A-T3 (0.064-IN. CLAD SHEET, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
40000 psi, min	45400	44300	46700	46700	46000	44600
	45100	44000	46200	50000	45400	44900
	44300	42800	46200	47200	44000	43800
	43700	44100	47000	48900	45400	44700
Average.	44600	43800	46500	48200	45200	44500
TENSILE STRENGTH, PSI						
62000 psi, min	67500	67900	66000	67400	67100	65300
	67200	68200	65700	66900	66700	65700
	67000	67500	65600	66000	64600	64600
	66000	66900	66500	67200	66800	65500
Average.	66900	67600	66000	66900	66300	65300
ELONGATION IN 2 IN., PER CENT						
15.0 per cent, min	21.0	20.0	20.5	19.0	18.0	18.0
	19.5	20.0	19.0	20.0	20.0	18.0
	20.0	20.0	20.0	20.0	20.0	19.0
	19.0	17.0	20.0	19.0	20.0	19.0
Average.	19.9	19.2	19.9	19.5	19.5	18.5

TABLE X. - DATA ON ALUMINUM ALLOY NO. ZG62A-T6 (0.064-IN. CLAD SHEET, PANEL).

ASTM Specification Requirements	3 YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT, PSI						
62000 psi, min	66700	69600	69900	72000	63500	70800
	66200	71000	68000	72200	65000	69900
	65300	71200	67500	71400	64000	70400
	66300	72200	69800	71800	64800	71700
Average.	66100	71000	68800	71800	64300	70700
TENSILE STRENGTH, PSI						
72000 psi, min	77600	82000	78100	80500	75600	76800
	79100	82000	76400	79300	76500	76900
	77900	83500	75900	78700	76000	77400
	77900	82400	77200	79300	76900	78100
Average.	78100	82500	76900	79400	76200	77300
ELONGATION IN 2 IN., PER CENT						
8.0 per cent, min	10.5	10.0	12.0	10.7	10.0	10.0
	10.5	9.5	11.0	10.5	11.0	10.0
	10.5	9.5	12.0	10.0	11.0	10.3
	10.5	9.5	11.0	10.0	10.0	11.0
Average.	10.5	9.6	11.5	10.3	10.5	10.3

TABLE XI. - DATA ON ALUMINUM ALLOY NO. GS11A-T6 (0.25-IN. PLATE, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
35000 psi, min	43000	40400	42700	41000	43000	40700
	42600	40400	42500	41900	43200	40300
	42900	41500	42400	41500	42800	42000
	42500	41900	42200	40900	43300	41600
Average.	42800	41000	42400	41300	43000	41200
TENSILE STRENGTH, PSI						
42000 psi, min	47100	46300	46600	46800	46800	45900
	47000	46100	46200	46600	46900	45900
	46900	46300	46100	46600	46600	46300
	47100	46300	46400	47000	46700	45900
Average.	47000	46200	46300	46800	46800	46000
ELONGATION IN 2 IN., PER CENT						
10.0 per cent, min	16.0	16.5	17.5	16.5	15.5	16.0
	17.0	16.0	17.0	16.0	15.5	15.0
	17.0	16.5	17.0	16.0	15.5	15.0
	17.5	17.0	17.0	16.0	15.5	16.0
Average.	16.9	16.6	17.1	16.1	15.5	15.5

TABLE XII. - DATA ON ALUMINUM ALLOY NO. CS41A-T4 (0.25-IN. PLATE, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
No Requirement	43500	45500	44200	44800	48800	44600
	43200	44500	43700	45300	47900	49300
	43500	46600	45000	45400	44200	50700
	43300	46700	44400	46100	46600	46500
Average.	43400	45800	44300	45400	46900	47800
TENSILE STRENGTH, PSI						
No Requirement	66800	66800	65400	66700	64500	65600
	66900	66800	65700	66900	64500	65300
	66800	67200	65200	66300	65100	64800
	66700	67100	65300	66400	64700	65300
Average.	66800	67000	65400	66600	64700	65200
ELONGATION IN 2 IN., PER CENT						
No Requirement	18.5	18.5	16.5	15.0	12.5	14.0
	18.5	19.0	16.0	16.0	13.0	12.0
	18.5	17.0	16.0	16.0	15.0	13.0
	18.5	18.0	16.5	16.0	13.5	11.0
Average.	18.5	18.1	16.2	15.8	13.5	12.5

TABLE XIII. - DATA ON ALUMINUM ALLOY NO. CS41A-T6 (0.25-IN. PLATE, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
No Requirement	66500	65700	66400	65200	64500	67000
	67100	65900	66700	65300	65800	67000
	66600	66300	65300	65300	65200	65600
	66600	66100	67400	67400	63800	67200
Average.	66700	66000	66400	65800	64800	66700
TENSILE STRENGTH, PSI						
No Requirement	72000	71400	70800	71400	68900	70700
	71900	71500	71500	70700	69100	70500
	71600	71700	70700	72100	69000	70700
	72000	72600	71400	70900	68100	70900
Average.	71900	71800	71100	71300	68800	70700
ELONGATION IN 2 IN., PER CENT						
No Requirement	10.5	9.0	8.0	9.0	6.0	8.0
	10.0	9.0	7.5	6.0	6.0	7.0
	10.0	9.0	8.0	7.0	6.0	7.0
	10.0	9.5	8.5	7.5	6.0	6.0
Average.	10.1	9.1	8.0	7.4	6.0	7.0

TABLE XIV. - DATA ON ALUMINUM ALLOY NO. CS41A-T6 (0.25-IN. CLAD PLATE, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
57000 psi, min	63400	62300	61000	60300	62400	61800
	63200	61500	62000	60700	62700	62900
	64200	61900	62100	61100	61500	62500
	62700	62400	61700	59800	63000	62600
Average.	63400	62000	61700	60500	62400	62400
TENSILE STRENGTH, PSI						
64000 psi, min	67300	67300	66600	67800	67000	66400
	67000	67500	66300	67200	66300	67100
	67400	67700	66800	67400	66300	67100
	67500	67400	67000	67100	66400	67100
Average.	67300	67500	66700	67400	66500	66900
ELONGATION IN 2 IN., PER CENT						
8.0 per cent, min	10.5	10.0	10.0	9.0	9.0	9.0
	10.0	10.5	8.0	9.0	9.0	8.0
	9.5	9.0	9.5	9.5	9.0	8.0
	9.5	9.6	10.0	9.5	9.0	8.0
Average.	9.9	9.8	9.4	9.2	9.0	8.2

TABLE XV. - DATA ON ALUMINUM ALLOY NO. CG42A-T4 (0.25-IN. CLAD PLATE, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
40000 psi, min	45000	47400	44500	47800	49800	50000
	44800	46000	44600	46100	47800	49600
	45100	45600	44400	47200	46800	52300
	44500	41600	44000	45600	49400	53300
Average. . . .	44800	45200	44400	46700	48700	51300
	TENSILE STRENGTH, PSI					
62000 psi, min	66000	67000	65400	68000	66300	65400
	66800	66100	65200	67000	66400	66100
	66900	65800	65500	67100	66300	65600
	66300	66500	65700	67400	66400	65300
Average. . . .	66500	66400	65400	67400	66400	65600
	ELONGATION IN 2 IN., PER CENT					
11.0 per cent, min	17.5	20.0	21.5	19.5	17.5	18.0
	19.0	19.5	19.5	21.0	16.5	18.0
	18.5	19.5	18.0	19.5	16.5	18.0
	18.5	19.0	18.0	18.0	16.5	18.0
Average. . . .	18.4	19.5	19.2	19.5	16.8	18.0

TABLE XVI. - DATA ON ALUMINUM ALLOY NO. ZG62A-T6 (0.25-IN. CLAD PLATE, PANEL).

ASTM Designation Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
62000 psi, min	68400	73300	69600	69800	70200	72800
	68200	74300	72200	72000	71200	70000
	68300	73300	72600	71300	71700	75600
	68800	72100	73200	70200	74100	74100
Average. . . .	68400	73200	71900	70800	71800	73100
	TENSILE STRENGTH, PSI					
72000 psi, min	77900	77700	77300	77900	76800	76900
	77800	77700	77200	77700	78700	77300
	77400	77200	77000	77600	76500	77400
	77600	77700	77200	77900	76800	76600
Average. . . .	77700	77600	77200	77800	77200	77000
	ELONGATION IN 2 IN., PER CENT					
8.0 per cent, min	9.0	11.0	10.0	9.0	9.0	9.0
	9.5	10.5	10.5	9.0	9.0	9.0
	9.0	10.0	10.5	9.0	9.0	9.0
	9.0	10.5	10.0	9.0	9.0	9.0
Average. . . .	9.1	10.5	10.2	9.0	9.0	9.0

TABLE XVII. - DATA ON ALUMINUM ALLOY NO. GS11A-T6 (0.25-IN. EXTRUDED BAR, PANEL).

ASTM Specification Requirements[a]	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
35000 psi, min	41400	41500	40000	41000	42300	40800
	41400	41500	39500	40200	41100	41000
	41100	41300	39700	40300	41100	40800
	41000	41300	39800	—	41200	41100
Average.	41200	41400	39800	40500	41400	40900
TENSILE STRENGTH, PSI						
38000 psi, min	44200	43900	43300	43400	43600	43400
	43900	43900	42700	42600	43400	43200
	43600	44000	46700	43100	43400	42600
	43800	44300	43000	43400	43700	43400
Average.	43900	44000	43900	43100	43500	43200
ELONGATION IN 2 IN., PER CENT						
10.0 per cent, min	17.0	16.5	15.5	16.5	15.0	14.0
	17.0	16.0	16.5	16.5	15.0	14.0
	16.5	16.0	16.0	15.0	14.0	14.0
	16.0	15.5	16.5	15.0	15.0	14.0
Average.	16.6	16.0	16.1	15.8	14.8	14.0

[a] Tentative Specifications for Aluminum-Alloy Extruded Bars, Rods and Shapes (B221-56T)

TABLE XVIII. - DATA ON ALUMINUM ALLOY NO. GS10A-T5 (0.25-IN. EXTRUDED BAR, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
16000 psi, min	24400	23800	23000	25200	26400	25400
	24800	23800	23500	24700	26500	25000
	24800	24200	24300	24800	26200	25300
	24300	24000	22900	25000	26700	24200
Average.	24600	24000	23400	24900	26400	25000
TENSILE STRENGTH, PSI						
22000 psi, min	28700	28200	28100	30200	29900	28600
	29300	28600	28400	29700	30500	29000
	29100	28900	28600	29800	29400	29000
	29000	28700	27300	29400	29200	29300
Average.	29000	28600	28100	29800	29800	29000
ELONGATION IN 2 IN., PER CENT						
8.0 per cent, min	17.5	17.0	17.0	15.0	14.5	15.0
	16.5	16.5	16.5	15.0	14.5	16.0
	17.0	17.0	16.5	16.0	14.5	16.0
	17.0	16.5	16.5	15.0	15.0	16.0
Average.	17.0	16.8	16.6	15.2	14.6	15.8

TABLE XIX. - DATA ON ALUMINUM ALLOY NO. CS41A-T4 (0.25-IN. EXTRUDED BAR, PANEL)

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
35000 psi, min	41500	41400	40300	43900	—	41000
	43800	43500	44300	43100	42200	44400
	45600	44000	43000	42100	40500	44100
	44200	43000	43000	39900	40000	44300
Average.	43800	43000	42100	42600	40900	43400
TENSILE STRENGTH, PSI						
50000 psi, min	57000	56600	56900	59000	57100	55400
	58500	60200	58700	58200	55000	58200
	59000	60500	59500	57600	54800	57100
	59200	59500	59300	56800	53700	57900
Average.	58400	59200	58600	57900	55200	57200
ELONGATION IN 2 IN., PER CENT						
12.0 per cent, min	27.0	23.5	25.5	24.0	20.0	22.0
	25.0	20.5	22.0	22.0	19.0	24.0
	25.0	20.5	21.0	24.0	19.0	20.0
	24.0	22.0	21.0	27.0	19.0	22.0
Average.	25.2	21.6	22.4	24.2	19.2	22.0

TABLE XX. - DATA ON ALUMINUM ALLOY NO. CS41A-T6 (0.25-IN. EXTRUDED BAR, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
53000 psi, min	60200	59000	59500	60900	61200	60100
	62800	62200	61700	61600	61000	63100
	63000	62800	62500	61000	60900	62200
	62300	62600	63000	58900	58800	61800
Average.	62100	61600	61700	60600	60500	61800
TENSILE STRENGTH, PSI						
60000 psi, min	63900	63100	62800	65400	63900	63500
	66300	66000	64600	65700	63700	65700
	66900	66900	65400	65400	63500	65000
	65800	66500	65900	62600	61900	65200
Average.	65700	65600	64700	64800	63200	64800
ELONGATION IN 2 IN., PER CENT						
7.0 per cent, min	15.0	12.5	13.0	10.0	8.0	10.0
	13.0	10.5	11.5	11.0	9.0	10.0
	13.0	10.5	11.5	11.0	9.0	10.0
	13.0	11.5	11.0	10.5	9.0	10.0
Average.	13.5	11.2	11.8	10.6	8.8	10.0

TABLE XXI. - DATA ON ALUMINUM ALLOY NO. ZG62A-T6 (0.25-IN. EXTRUDED BAR, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
72000 psi, min	88800 85100 81700 84300	84600 85400 83500 84300	85800 80300 79500 82500	82400 78500 82800 86200	80100 81500 83100 82300	86100 85100 84300 81100
Average.	85000	84400	83000	82500	81800	84200
TENSILE STRENGTH, PSI						
80000 psi, min	94400 90900 88800 90200	88600 89200 87300 87200	93000 87900 86600 89000	98700 85600 88300 92700	86800 88700 88100 91600	92100 89800 90300 87100
Average.	91100	88100	89100	91300	88800	89800
ELONGATION IN 2 IN., PER CENT						
7.0 per cent, min	9.5 10.5 9.0 9.5	10.5 10.0 11.0 9.0	9.0 8.0 8.0 8.5	8.0 9.0 7.0 9.0	6.0[a] 9.0 7.0 7.0	8.0 8.0 8.0 7.0
Average.	9.6	10.1	8.4	8.2	7.3	7.8

[a] LOW

TABLE XXII. - DATA ON ALUMINUM ALLOYS NOS. GS11A-T6, CS41A-T6, CG42A-T4 AND ZG62A-T6 (0.064-IN. SHEET, SPOT WELD[a]).

	BREAKING LOAD, LB					
	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	ALLOY NO. GS11A-T6					
	1650 1615 1605 1670	1560 1560 1535 1555	1580 1515 1500 1730	1500 1475 1455 1580	1340 1920 750 1955	No Value
Average.	1635	1552	1581	1502	1491	
	ALLOY NO. CS41A-T6 - CLAD					
	1590 1795 1625 1955	2400 1957 1835 1915	2010 1660 1895 1755	1995 1780 1585 1890	1560 2430 -- --	No Value
Average.	1741	2027	1830	1812	1995	
	ALLOY NO. CG42A-T4 - CLAD					
	2070 1745 2285 2235	1800 2500 2165 1675	1650 1860 2120 1915	1860 1500 1950 1915	2195 1765 1935 —	No Value
Average.	2084	2035	1886	1806	1965	
	ALLOY NO. ZG62A-T6 - CLAD					
	2510 2355 2370 2405	2280 2670 2660 2580	2055 2155 2020 2185	2860 2390 2360 2500	1750 1565 1690 1690	2705 2686 2518 2124
Average.	2410	2548	2104	2528	1674	2508

[a] Values are shear results for 2 spot welds on a single lap joint.

TABLE XXIII. - DATA ON ALUMINUM ALLOYS NOS. GS11A-T6 AND GS10A-T5.

	TENSILE STRENGTH, PSI					
	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	ALLOY NO. GS11A-T6 (0.25-IN. PLATE, GAS WELD)					
	20800[a]	21800	22700	No	21400	26700
	22100	22000	22700	Value	21200	26100
	22100	22100	23100		21300[a]	26100
	22100	22000	23100		19200[a]	25800
Average.	21800	22000	22900		20800	26200
	ALLOY NO. GS11A-T6 (0.25-IN. PLATE, ARC WELD)					
	22500	24200	22600[a]	No	22700[a]	22000
	25700	24000	22600[a]	Value	21000[a]	22800
	25600[a]	24700	25400		25200[a]	22700
	25600	24300	25400		20300[a]	21400
Average.	24800	24300	24000		22300	22200
	ALLOY NO. GS10A-T5 (0.25-IN. EXTRUDED BAR, ARC WELD)					
	20500	21400	21600	20500	20700	20400
	21000	21300	21600	20900	20900	20900
	21000	21400	21900	20900	20900	20500
	20900	21100	20900	20500	20400	20900
Average.	20800	21300	21500	20700	20700	20700

[a] Samples failed through weld.
All other samples failed in base material.

TABLE XXIV. - DATA ON ALUMINUM ALLOYS, NOS. GS11A-T6, CG42A-T3, AND ZG62A-T6 (0.064-IN. SHEET, RIVET[a, b])

	TENSILE STRENGTH, PSI					
	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
		ALLOY NO. GS11A-T6				
	46900	47100	45000	44100	44900	47000
	47000	48500	45200	44500	45800	47000
	46600	48500	44400	46000	44500	46400
	45600	48700	45400	45400	44300	46800
	46400	48400	45200	46200	44900	46500
	45000	48500	45400	45300	43000	47200
Average.	46200	48300	45100	45200	44600	46800
		ALLOY NO. CG42A-T3				
	62200	64900	63400	63000	48700[c]	59700
	64600	64800	63600	62200	51700[c]	62000
	60500	62100	63300	64300	—[c]	62000
	63500	64200	63000	64600	—[c]	62400
	64700	65400	64700	64000	—[c]	61800
	—	—	—	60900	—[c]	59500
Average.	63100	64300	63600	63200	50200	61200
		ALLOY NO. ZG62A-T6				
	78000	78000	79900	79800	78100	80400
	79100	77800	79100	77900	78100	80200
	78500	78000	78900	78800	78400	80200
	78500	79100	78600	78600	77800	80900
	78400	78800	78000	78600	75400	80900
	78000	79500	77800	80800	79200	80700
Average.	78400	78500	78700	79100	77800	80600

[a] Net area = Width - diameter of rivet hole x thickness.
[b] All specimens failed in sheet.
[c] Samples badly corroded.

TABLE XXV. - DATA ON ALUMINUM ALLOYS, NOS. CS41A-T6, CG42A-T4, AND ZG62A-T6 (0.064-IN. CLAD SHEET, RIVET^{a,b}).

	TENSILE STRENGTH, PSI					
	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
		ALLOY NO. CS41A-T6				
	67800	66700	66500	67200	67100	69500
	67300	66900	67300	68300	66000	67800
	67800	66200	65900	67600	66200	68900
	67600	66700	66200	67600	66400	69300
	65900	66700	65700	66000	66400	69100
	67700	67400	66700	67500	68500	68400
Average.	67400	66800	66400	67400	66800	68800
		ALLOY NO. CG42A-T3				
	62900	61500	61500	60600	62200	60500
	61500	64100	64100	62200	61800	57800
	61500	64800	64800	63700	60900	63400
	63600	64200	64300	62200	59500	60300
	62100	64600	64600	63700	62400	58600
	62300	—	—	63100	59500	62800
Average.	62300	63800	63900	62600	61000	60600
		ALLOY NO. ZG62A-T6				
	74700	77400	73700	66600	74600	80000
	74900	79100	75700	73300	73100	78600
	73200	77800	75300	74200	74200	78100
	75300	76700	75500	73500	71200	78600
	74700	77400	75700	78500	74400	78800
	73000	77400	75300	75300	76000	77400
Average.	74300	77600	75200	73600	73900	78600

[a] Net area = Width - diameter of rivet hole x thickness.
[b] All specimens failed in sheet.

TABLE XXVI. - DATA ON MAGNESIUM ALLOY NO. M1A-O (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements[a]	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
No Requirements	16800 17200 17500 17100	15900 16000 16100 16100	13500 14000 14800 12500	15200 15300 15000 15000	16000 16200 15600 16100	16100 16400 16200 16000
Average.	17200	16000	13700	15100	16000	16200
	TENSILE STRENGTH, PSI					
38000 psi, max[c]	32400 32600 32600 32800	28200 28200 28200 28200	25700 25700 26500 26400	27300 28100 27900 27300	28100 27900 27800 27800	29500 30100 28500 29600
Average.	32600	28200	26100	27600	27900	29400
	ELONGATION IN 2 IN., PER CENT					
12.0 per cent, min	18.0 19.0 14.0 18.0	6.5^b 7.0^b 7.0^b 7.0^b	13.0 12.0 15.0 13.0	5.5^b 6.5^b 6.0^b 5.0^b	7.0^b 6.0^b 5.5^b 5.5^b	6.5^b 7.0^b — 7.5^b
Average.	17.2	6.9^b	13.2	5.8^b	6.0^b	7.0^b

[a] Tentative specifications for magnesium-base alloy sheet (B90-56T).
[b] LOW
[c] To insure complete annealing

TABLE XXVII. - DATA ON MAGNESIUM ALLOY NO. AZ31A-H24 (0.064-IN. SHEET, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
29000 psi, min	29800	28500^a	29300	29400	29700	29600
	28900^a	28300^a	29700	29600	29600	30000
	29800	28700^a	29700	29900	29400	29400
	30000	28300^a	29500	29700	30200	29700
Average.	29600	28400^a	29600	29600	29700	29700
		TENSILE STRENGTH, PSI				
39000 psi, min	42300	39200	36500^a	41000	39200	42500
	42000	39000	37100^a	41000	39200	42500
	41600	39300	36900^a	41000	40500	42400
	41600	39000	36900^a	41000	41000	42500
Average.	41900	39100	36800^a	41000	40000	42500
		ELONGATION IN 2 IN., PER CENT				
4.0 per cent, min	17.0	8.0	9.0	11.0	8.0	8.5
	14.0	8.5	11.0	8.5	8.0	9.0
	17.0	8.0	12.0	9.0	7.5	10.5
	9.0	8.5	9.0	9.5	7.5	9.5
Average.	14.2	8.2	10.2	9.5	7.8	9.4

^{a}LOW

TABLE XXVIII. - DATA ON MAGNESIUM ALLOY NO. AZ31A-H24 (0.25-IN. PLATE, PANEL).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
29000 psi, min	33100	32100	31300	32300	32100	31200
	32800	31900	31300	32300	32300	31500
	—	32100	31500	32400	31900	31300
	—	32000	—	32500	31700	31200
Average.	33000	32000	31400	32400	32000	31300
		TENSILE STRENGTH, PSI				
39000 psi, min	41900	41200	40600	41600	41200	41800
	41900	41100	40700	41600	40700	41700
	41900	41100	40600	41500	40900	41800
	41900	41200	40300	41500	41100	41700
Average.	41900	41200	40600	41600	41000	41800
		ELONGATION IN 2 IN., PER CENT				
4.0 per cent, min	15.0	11.5	14.0	9.0	6.5	13.0
	16.0	11.5	14.0	10.0	6.5	13.0
	14.8	11.5	11.5	10.0	6.5	12.0
	18.0	12.0	9.5	10.0	10.0	12.5
Average.	16.0	11.6	12.2	9.8	7.4	12.6

TABLE XXIX. - DATA ON MAGNESIUM ALLOY NO. MIA-F (0.25-IN. EXTRUDED BAR).

ASTM Specification Requirements[a]	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
	23000 21700 21800 23700	— 21900 22000 22000	23200 22900 22800 —	23700 22300 22300 23600	21900 23800 21900 23800	20600 21500 21400 20300
Average.	22600	22000	23000	23000	22800	21000
TENSILE STRENGTH, PSI						
32000 psi, min	33400 33300 33300 33600	31100[b] 31400[b] 31200[b] 31500[b]	31900[b] 31900[b] 31900[b] 31900[b]	32800 31900[b] 32200 32300	31900[b] 33000 32100 32600	31600[b] 32900 32500 32400
Average.	33400	31300[b]	31900[b]	32300	32400	32400
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min	14.0 10.0 10.0 12.0	— 5.5 5.0 5.5	10.5 10.0 9.5 9.5	6.0 5.5 6.0 6.0	5.0 6.0 6.0 5.5	5.0 6.0 5.5 5.5
Average.	11.5	5.3	10.0	5.9	5.6	5.5

[a] Tentative specifications for magnesium-base alloy bars, rods, and shapes (B107-56T).

[b] LOW

TABLE XXX. - DATA ON MAGNESIUM ALLOY NO. AZ31B-F (0.25-IN. EXTRUDED BAR).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
20000 psi, min	24800 24800 25000 —	25100 24900 25100 25100	25400 25800 25400 25200	25700 25700 25700 25700	24900 24900 24900 24800	24500 24600 24600 24600
Average.	24900	25000	25400	25700	24900	24600
TENSILE STRENGTH, PSI						
32000 psi, min	38500 38800 38800 38400	37900 37900 37900 37900	36800 36600 36700 36900	37500 37600 37600 37600	37400 37700 37600 37700	38000 38000 37800 38300
Average.	38600	37900	36800	37600	37600	38000
ELONGATION IN 2 IN., PER CENT						
7.0 per cent, min	14.0 16.5 16.5 16.0	14.5 13.5 15.0 15.0	13.5 14.5 13.5 13.5	11.5 15.5 12.5 15.0	14.5 11.0 14.0 12.5	13.5 15.0 15.0 14.0
Average.	15.8	14.5	13.8	13.6	13.0	14.4

TABLE XXXI. - DATA ON MAGNESIUM ALLOY NO. AZ80A-T51 (0.25-IN. EXTRUDED BAR).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
32000 psi, min	39100	38600	37700	39100	36500	37600
	37900	38400	38200	38900	37400	38400
	38700	38500	37600	38800	36500	37300
	39200	38800	37600	38900	36600	37500
Average.	38700	38600	37800	38900	36800	37700
	TENSILE STRENGTH, PSI					
47000 psi, min	54200	52400	51900	51300	49000	51500
	53300	52100	51700	50800	50400	51400
	53400	52100	51000	51300	50000	50100
	53400	52100	51600	51400	49300	50600
Average.	53600	52200	51600	51200	49700	50900
	ELONGATION IN 2 IN., PER CENT					
3.0 per cent, min	8.5	6.5	5.5	4.5	5.0	5.0
	11.0	7.0	5.0	4.0	6.0	5.0
	10.0	7.0	6.0	4.5	5.0	—
	10.0	6.5	6.5	4.0	5.0	5.0
Average.	9.9	6.8	5.8	4.2	5.2	5.0

TABLE XXXII. - DATA ON MAGNESIUM ALLOY NO. ZK60A-F (0.25-IN. EXTRUDED BAR).

ASTM Specification Requirements	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
31000 psi, min	36400	35200	34700	37400	36500	36600
	36200	35200	34900	37500	37400	36900
	35900	34900	—	37600	36500	36600
	36400	35100	—	37500	36600	36200
Average.	36200	35100	34800	37500	36800	36600
	TENSILE STRENGTH, PSI					
43000 psi, min	47100	45500	44800	47600	45000	47500
	47200	45400	44900	47700	46000	47500
	47600	45300	44900	47700	45500	47700
	47100	45500	45200	47600	45500	47500
Average.	47200	45400	45000	47600	45500	47600
	ELONGATION IN 2 IN., PER CENT					
5.0 per cent, min	8.5	8.5	14.0	11.5	10.5	9.5
	10.5	9.0	11.0	11.5	10.0	9.5
	10.0	8.5	11.0	10.0	—	10.0
	10.0	8.0	12.0	11.0	10.5	10.0
Average.	10.0	8.5	12.0	11.0	10.3	9.6

TABLE XXXIII. - DATA ON MAGNESIUM ALLOYS NOS. M1A-0, AZ31A-H24 (0.064-IN. SHEET, SPOT WELD).

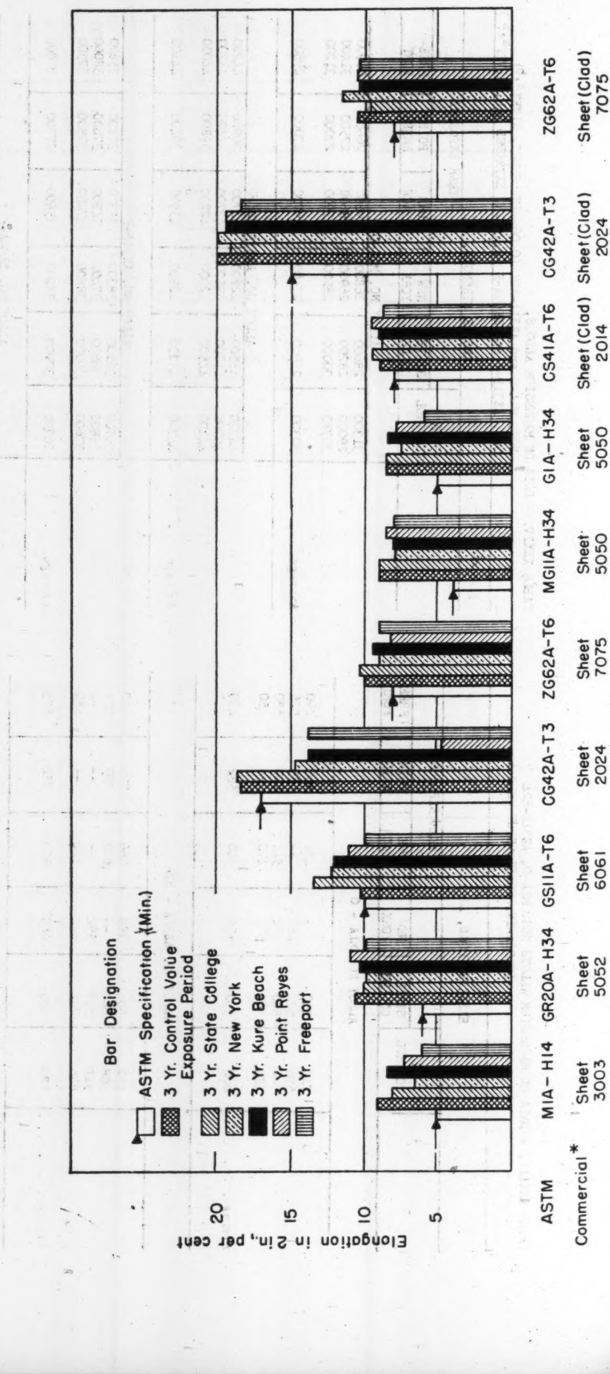
	BREAKING LOAD, LB					
	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
ALLOY NO. M1A - 0						
	744	430	--	486	560	490
	596	504	468	—	685	525
	--	500	472	544	500	530
	484	568	576	470	—	450
Average.	608	500	505	500	582	499
ALLOY NO. AZ31A - H24						
	592	504	780	766	775	650
	508	668	—	678	790	755
	544	512	592	—	—	--
	676	732	624	592	—	650
Average.	580	604	665	679	782	685

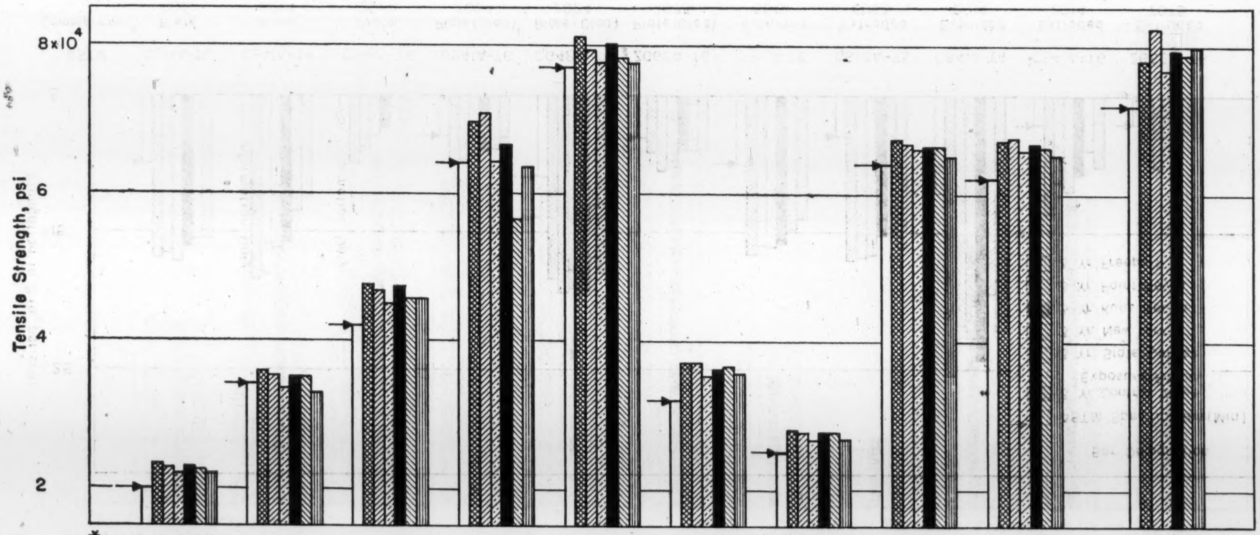
Values are shear results for 2 spot welds on a single lap joint.

TABLE XXXIV. - DATA ON MAGNESIUM ALLOYS,
NOS. M1A-0 and AZ31A-H24 (0.064-IN. SHEET, RIVET[a,b])
NOS. AZ31B-F and ZK60A-F (0.25-IN. EXTRUDED BAR, RIVET[a,b])

	TENSILE STRENGTH, PSI					
	3-YR CONTROL	3 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
ALLOY NO. M1A-0						
	31300	28600	26800	29600	26600	26900
	29000	28200	26900	29600	27500	31400
	30700	30000	28500	29900	27000	31200
Average. . . .	30300	28900	27400	29700	27000	29800
ALLOY NO. AZ31A-H24						
	44300	45500	42800	44700	36900	44200
	45700	45700	37500	43700	41400	44200
	44100	44500	42000	42200	39800	44000
Average. . . .	44700	45200	40800	43500	39400	44100
ALLOY NO. AZ31B-F						
	36700	38100	38300	35300	37800	35600
	35800	36600	37400	34200	37400	38000
	37600	39000	38800	37400	37800	37500
Average. . . .	36700	37900	38200	35600	37700	37000
ALLOY NO. ZK60A-F						
	41600	43200	44100	45700	42500	43600
	45000	46400	45000	44300	45000	43500
	44600	45700	45200	45000	42700	45800
Average. . . .	43700	45100	44800	45000	43400	44300

[a] Net area = width - diameter of rivet hole × thickness.
[b] All specimens failed in sheet.

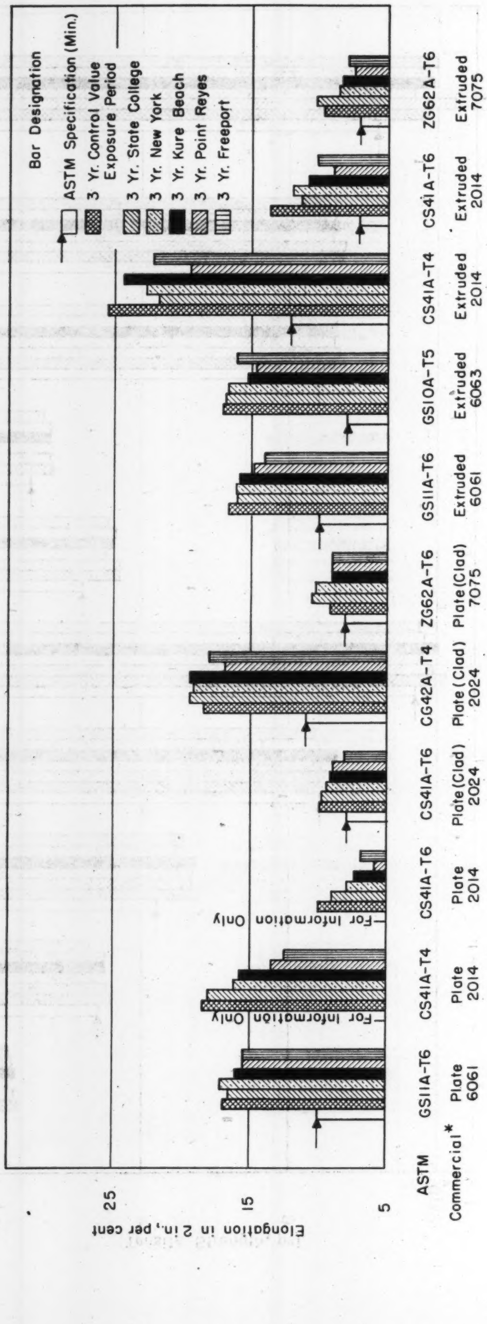

Bar Designation
ASTM Specification (Min.)
3 Yr. Control Value Exposure Period
3 Yr. State College
3 Yr. New York
3 Yr. Kure Beach
3 Yr. Point Reyes
3 Yr. Freeport
Elongation in 2 in., per cent
5
10
15
20
ASTM
Commercial*
M1A-H14 Sheet 3003
GR20A-H34 Sheet 5052
GS11A-T6 Sheet 6061
CG42A-T3 Sheet 2024
ZG62A-T6 Sheet 7075
MG11A-H34 Sheet 5050
G1A-H34 Sheet 5050
CS41A-T6 Sheet (Clad) 2014
CG42A-T3 Sheet (Clad) 2024
ZG62A-T6 Sheet (Clad) 7075



*As Most Commonly Used

Each Bar Represents Average Results on Four Specimens

FIG. 1.—Summary of Data, Atmospheric Exposure of Aluminum Alloy Panels 0.064 in.


Bar Designation
ASTM Specification (Min.)
3 Yr. Control Value Exposure Period
3 Yr. State College
3 Yr. New York
3 Yr. Kure Beach
3 Yr. Point Reyes
3 Yr. Freeport
Elongation in 2 in., per cent
25
15
5
ASTM
Commercial*
GS11A-T6
Plate 6061
CS41A-T4
Plate 2014
CS41A-T6
Plate 2014
CS41A-T6
Plate (Clad) 2024
CG42A-T4
Plate (Clad) 2024
ZG62A-T6
Plate (Clad) 7075
GS11A-T6
Extruded 6061
GS10A-T5
Extruded 6063
CS41A-T4
Extruded 2014
CS41A-T6
Extruded 2014
ZG62A-T6
Extruded 7075
For Information Only
For Information Only

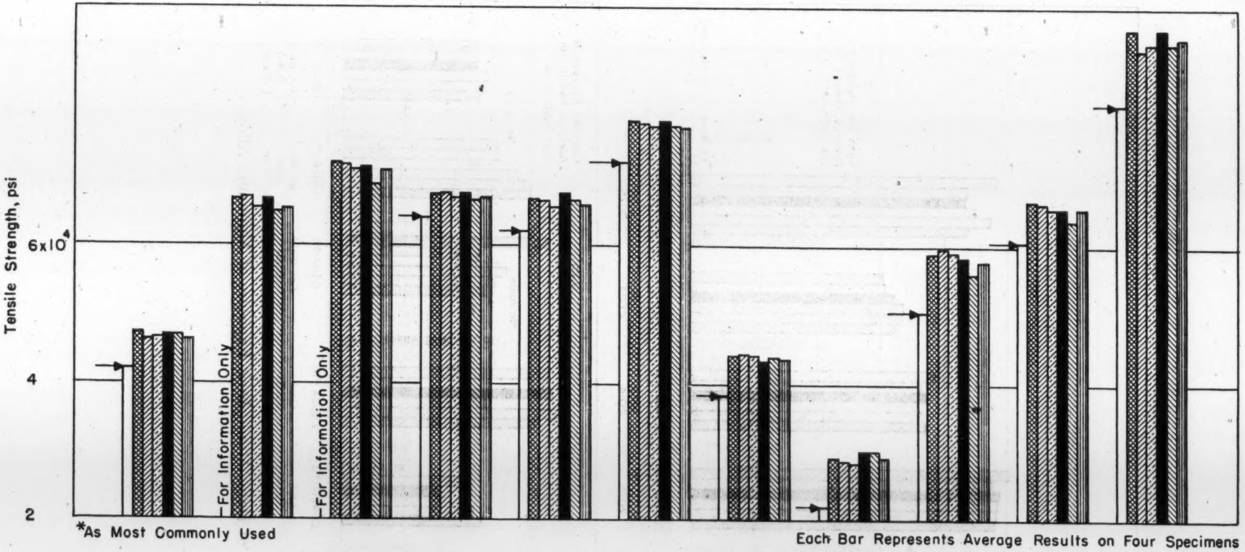


FIG. 2.—Summary of Data, Atmospheric Exposure of Aluminum Alloy Panels 0.25 in.

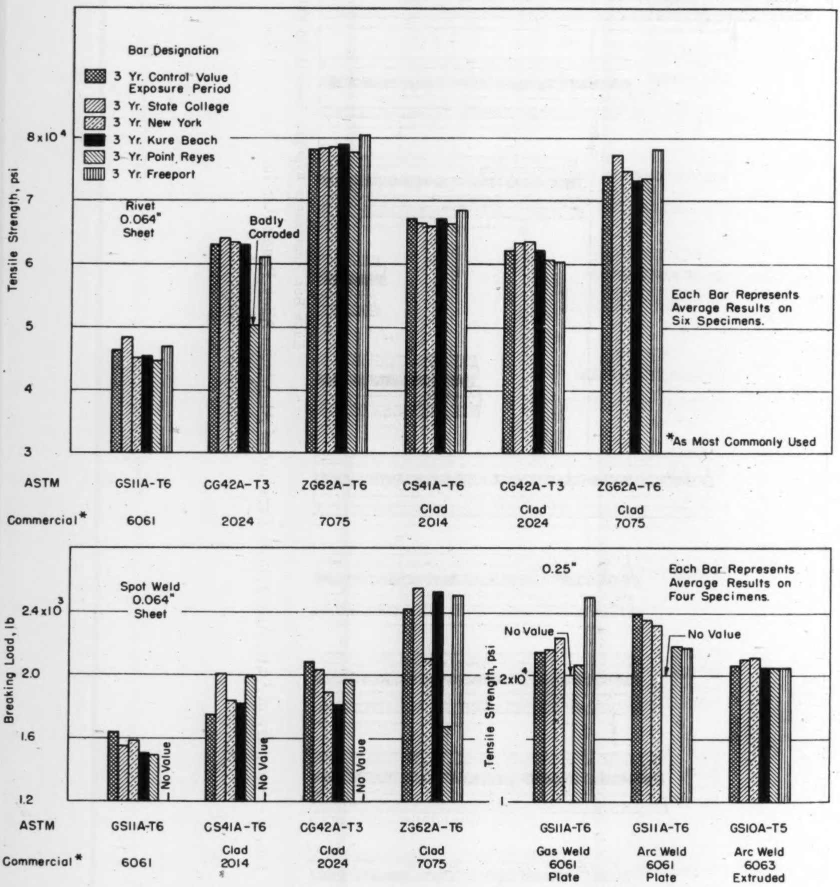
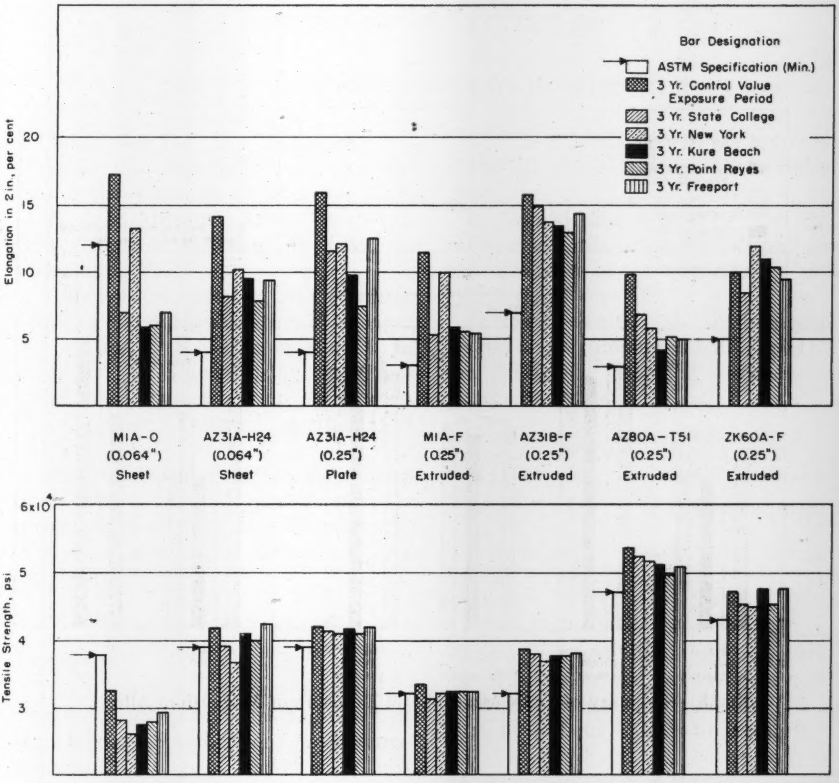


FIG. 3.—Summary of Data, Atmospheric Exposure of Aluminum Alloys.



Each Bar Represents Average Results on Four Specimens.

FIG. 4.—Summary of Data Atmospheric Exposure—Magnesium Alloy Sheet, Plate and Extruded Bars.

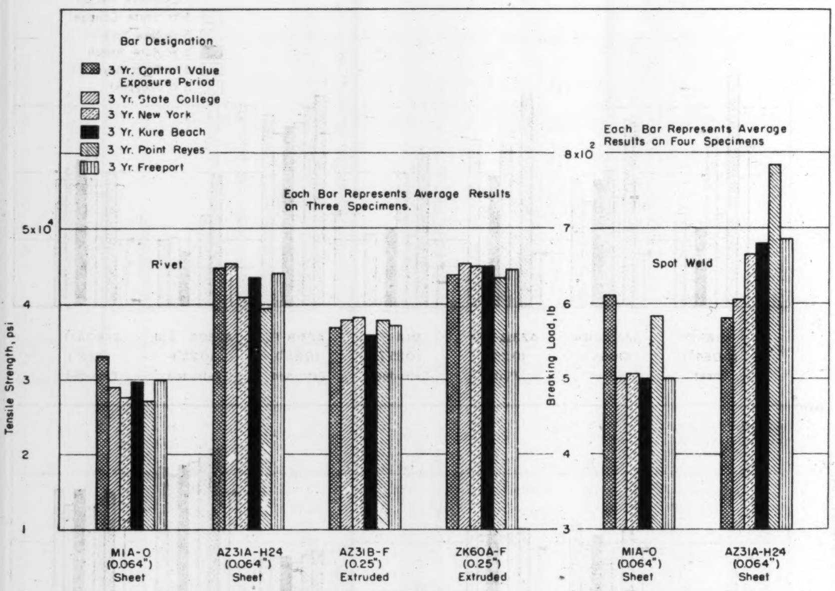


FIG. 5.—Summary of Data Atmospheric Exposure of Magnesium Alloys.

REPORT OF COMMITTEE B-8
ON
ELECTRODEPOSITED METALLIC COATINGS*

Committee B-8 on Electrodeposited Metallic Coatings held two meetings during the year: on October 23, 1958, at Battelle Memorial Inst., Columbus, Ohio, and on February 6, 1959, at Pittsburgh, Pa.

At its February meeting the committee voted to sponsor publication of a paper by Messrs. A. K. Graham and H. L. Pinkerton on "Standardizing the Preparation of Electrodeposits on Test Panels for Corrosion Testing" in the 1959 *Proceedings*.[1]

The membership of the committee totals 188, of whom 67 are classified as producers, 24 as consumers, 85 as general interest members, and 12 as consulting members.

Revision of Standard, Immediate Adoption

The committee recommends for immediate adoption revisions as follows of the Standard Specifications for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (**B 142 – 58**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Section 1.—Add a fourth type of coating, designated as type DZ.

In Note 2 insert "D" at the beginning of the list of initial letters in the second line.

In Note 3, line 4, insert "type DZ" before the words "type FZ." In the second paragraph of the note change "type FZ" to read "type DZ."

Section 3.—To the table in Paragraph (*a*) add a new column headed "Type DZ" and listing the following thickness requirements:

Copper plus nickel, min.	0.0020 in. (51 μ)
Copper, min.	0.00020 in. (5.1 μ)
Final nickel, min.	0.0010 in. (25 μ)
Chromium (if required), min.	0.000010 in. (0.25 μ)

In Paragraph (*b*), in the third line, insert the words "type DZ" before "type FZ."

Appendix.—Under the section on Copper and Nickel, in the second paragraph, line 9, following the words "per sq ft" insert the words "for 0.003 in. (76 μ) (type DZ) about 75 amp-hr per sq ft."

Editorial Change

The committee recommends editorial changes as follows in the Recommended Practice for Preparation of and Electroplating on Stainless Steel (**B 254 – 53**):[2]

Appendix.—Add a new Item (*3*) to Section A5(*f*) to read "(*3*) Electrolytic molten baths may also be employed," renumbering the present Item (*3*) as (*4*).

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] See p. 317.

[2] 1958 Book of ASTM Standards, Part 2.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications, Papers, and Definitions (F. Ogburn, chairman).—Section C (W. E. Moline, chairman) has prepared a monograph on electroless nickel, with publication expected during 1959.

A task group with D. M. Bigge as chairman recommended the revision of Specification B 142 by the addition of a fourth type of coating, as indicated earlier in this report.

Subcommittee II on Performance Tests (W. L. Pinner, chairman).—Section A (B. B. Knapp, chairman) is making a critical review of a report covering preparation and exposure of Program IV panels before resubmission to letter ballot.

Section E (F. A. Lowenheim, chairman) is testing solderability of tin, tin-zinc, and tin-lead coatings.

Section F (J. W. Kerstetter, chairman) is completing the copper-nickel-chromium plating of cast and wrought aluminum panels for exposure tests.

Subcommittee III on Conformance Tests (A. D. Squitero, chairman).—Section B (A. Mendizza, chairman) has outlined a program for comparing and correlating results from the following corrosion tests: Corrodkote, acetic acid salt spray, accelerated acetic acid (acetic acid salt spray with copper chloride added), and regular salt spray.

Subcommittee IV on Electroplating Practice (E. B. Saubestre, chairman).—Section N (B. Knapp, chairman) has prepared a proposed recommended practice for electroplating nickel on nickel, which will be submitted to letter ballot by Subcommittee IV. The subcommittee suggested the editorial change in Recommended Practice B 254 referred to earlier in the report.

Subcommittee V on Supplementary Treatments (G. E. Best, chairman).—Section B (A. Mendizza, chairman) is establishing a program to investigate the effect of storing untreated and chromate treated zinc plate in polyethylene bags prior to exposure testing, in order to explain unusual corrosion behavior of zinc-plated steel so treated and stored.

This report has been submitted to letter ballot of the committee, which consists of 188 voting members: 99 members returned their ballots, of whom 97 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
Chairman.

D. M. BIGGE,
Secretary.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

APPENDIX

STANDARDIZING THE PREPARATION OF ELECTRODEPOSITS ON TEST PANELS FOR CORROSION TESTING

By A. K. Graham[1] and H. L. Pinkerton[1]

During the past ten years, a great deal of thought and study has gone into the standardization of methods of rating plated samples corroded by atmospheric or accelerated testing (**1–8**).[2] The aim has been to describe the extent and nature of the failure in an understandable way and reproducibly so that valid comparisons may be made between the behavior of different test lots inspected by different teams.

Much progress has been made in this direction; unfortunately, however, too little attention has been paid to standardization at the other end of the process, namely, the production of the samples, or test panels, for such corrosion testing. Extensive programs have been laid out (**2–13**), carried through, and conclusions drawn, although control of the sample preparation is rarely reported in sufficient detail, has sometimes been demonstrably lacking, and has never been standardized in an officially accepted procedure.

The 1947 exposure corrosion test Program No. 1, sponsored by ASTM Subcommittee II on Performance Tests of Committee B-8 (**14–17**), was planned by a committee consisting of representatives of most of the bumper and automobile manufacturing companies, William Blum, and A. K. Graham. In the opinion of many and in the light of experience, it was particularly well-conceived with respect to the control of the uniformity of the test panels. The only published reports on this program appear in the Subcommittee II reports in the ASTM *Proceedings* (**14–17**). Furthermore, even these reports do not include certain procedural details which the authors feel are of the utmost importance. The procedures used in evaluating and standardizing the cleaning cycles were published later (**18**). It is the purpose of this paper to recommend the preparation of specifications for the plating of corrosion test panels and to suggest some of the basic requirements that should be a part of such specifications. To this end, a description of the procedures used in the 1947 corrosion test Program No. 1, and an analysis of some of the data obtained may be instructive.

Preliminary Work

A great deal of preliminary work was done to standardize not only procedural details but also methods of control. One valuable tool, however, was not utilized, namely, the application of statistical quality control methods, which were not widely appreciated at that time. As will be shown, analysis of the original data indicates that while a high degree of control was actually attained, it could have been further improved had these methods been employed.

An earnest attempt was made to recognize and control all variables that might have a significant bearing on corrosion resistance. It is convenient to

---

[1] President and Engineer, respectively, Graham, Savage and Associates, Inc., Jenkintown, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

classify these variables as follows:

(1) Basis metal condition and mechanical processing,

(2) Preparation for plating,

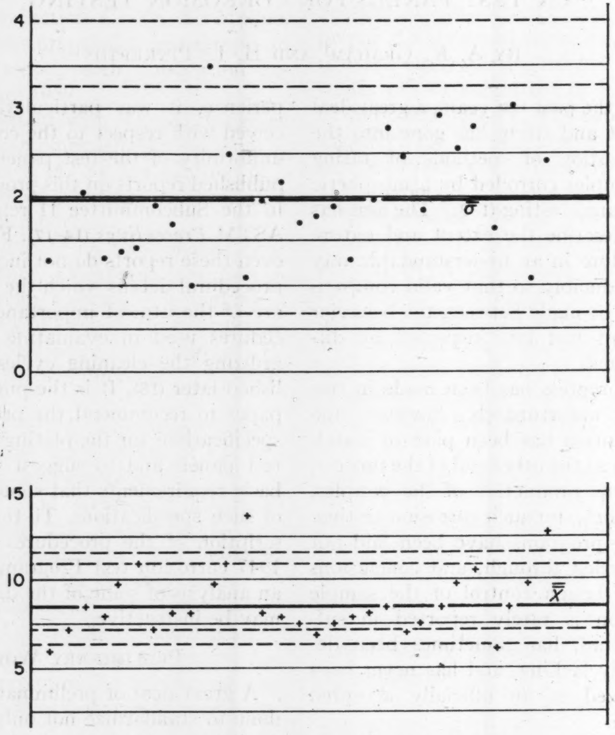


FIG. 1.—Profilometer Readings on Polished Steel Panels.

Lower graph.—Successive lot averages of 29 lots of 6 panels each, root-mean-square in microinches.

Upper graph.—Standard deviations of each lot, plotted around $\bar{\sigma}$, the standard deviation of the grand lot.

(3) Control of plating variables,

(4) Buffing of deposits, and

(5) Calibration of controls.

The philosophy of the entire program was to use procedures that were in line with good commercial practice but with controls that were, within reason, the most exacting that could be devised.

BASIS METAL PROCESSING

The plan called for using two different steels, one of which was to be heat treated. All steel was to be polished to what was considered an acceptable commercial finish, except that one lot was to be given a much better finish. A lot was to consist of 48 panels, cut to the standard 4 by 6-in. size. Each lot of steel was obtained from the same mill heat. Analyses of the heats were run on representative samples of each steel.

The sheets were sheared into 4 by 6-in. ($\pm\frac{1}{32}$ in.) panels, with the 6 in. axis of the panels always parallel to the long axis of the sheet. A $\frac{1}{4}$-in. hole was punched on the center line of a 4-in. edge $\frac{1}{4}$ in. from the edge, and the hole was countersunk on the face for a No. 10 machine screw, used for bolting the panel to the plating rack.

The panels were examined, and those with obvious steel defects were discarded. The remainder were stamped to identify run number, steel, and individual panel number. Those not requiring heat treatment were slushed with a rust preventative, allowed to dry, and stacked in storage boxes lined with waterproof paper. Compartmented boxes were provided for transporting panels through processing without damage. The lots requiring it were then heat treated, immediately straightened in a flattening die, air-cooled without stacking, and then stored in the sealed condition in similar storage boxes. These were not pickled until they were ready to be polished.

Preliminary work had shown that polishing on a wheel, besides being rather nonreproducible, destroyed the true flatness of the surface, so that machine-buffing after plating was very irregular, in many cases showing gradations from good buffing to no buff at all on the same panel. Consequently, polishing was done on a belt-and-backstand unit. Panels were held individually on wooden paddles applied by hand against the belts over a fulcrum. Two paddles were used, each having its guide strips so set that the panel was rotated 60 deg, in shifting from one paddle to the other. The panels were changed from one paddle to the other with every change of polishing grit. No set number of passes on each belt was specified. Instead, the operator gave the panel as many passes as necessary to obliterate previous grit marks, as determined by visual inspection, and then one final pass with the same belt before going to the next. The sequence of belts used had been standardized in preliminary work. A sequence of 100–120–150–180 grease was chosen, to obtain a steel finish which enabled the buffing machine to produce a good commercial finish on the nickel plate with minimum removal of deposited metal. Alumina abrasive belts were used; they were chosen over silicon carbide belts because the latter were judged to have too severe a cutting action.

Profilometer readings were taken on panels Nos. 1, 15, 20, 31, 38, and 45 from each lot of 48, representing one panel chosen at random from each rack load of 8. The data are shown in Fig. 1, plotted as for statistical quality control, in chronological order. Each six readings were taken as samples representing the lot of 48, and the standard deviations of each lot were plotted in the usual way around the uncorrected standard deviation of the grand lot, between limits for sample size 6. It will be seen that there are no extreme mavericks, and only the tenth lot is a suspected maverick. This is fewer than would be expected in 29 lots, so there is no cause to correct the standard deviation of the grand lot. However, when the average profilometer readings for each lot are plotted in the same way, there are three extreme mavericks and a number of suspected mavericks. Furthermore, it is evident that a regular change is taking place from lot to lot in a cyclic way, probably (in retrospect) having something to do with the replacing of worn belts with new ones. The fact that the standard deviations are under control, while the lot averages are not, indicates that the operator was doing a consistent job with tools that were changing in some regular way. (Note that about every sixth or seventh lot is high.)

If such control plots had been made

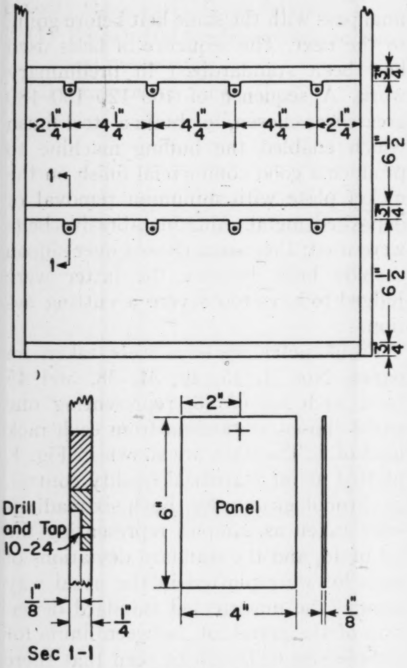

3/4"
2 1/4"
4 1/4"
4 1/4"
4 1/4"
2 1/4"
6 1/2"
1
3/4"
1
6 1/2"
3/4"
2"
Panel
6"
Drill and Tap 10-24
1/8"
1/4"
4"
1/8"
Sec 1-1

FIG. 2.—Plating Rack for Test Panels.

while the work was in progress, it should have been possible to correlate the gradual rise in root-mean-square from lot to lot with belt changes, or whatever else caused it. The result would have been a better (lower) average profilometer reading with less spread in the values because corrective measures could have been applied before the lot averages got out of control.

PREPARATION FOR PLATING

Since no variations in cleaning cycles were under study in the program, it was simply desired to use the best possible cycle and to control it adequately. It was reasoned that the best test for the thoroughness of a preplating cycle is the adhesion of the subsequent electrodeposit. Accordingly, the steps in the cleaning cycle were chosen to represent good practice. The specific conditions were then varied until a satisfactory degree of adhesion was attained.

The peel test (18) was used to evaluate adhesion and thus to select satisfactory cleaning cycles. In addition, the adhesion

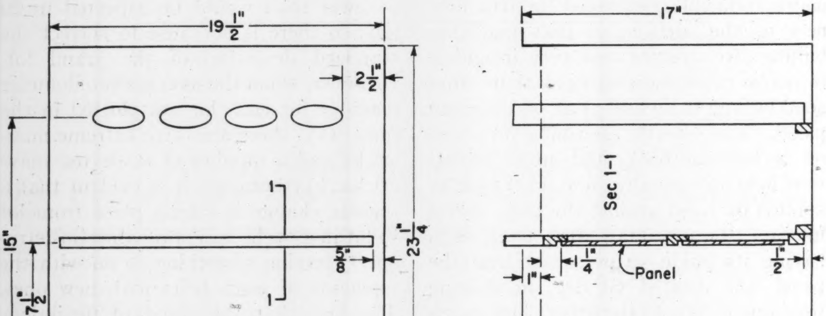

19 1/2"
2 1/2"
15"
7 1/2"
1
1
5/8"
23 1/4"
Y
X
2 kw Heater
17"
Sec 1-1
1"
1/4"
1/2"
Panel

FIG. 3.—Rack Arrangement in Plating Tank.

was also checked during production, before start of operations each Monday morning, or whenever the cycle had to be changed. Panels Nos. 1 and 31 from each lot, which were destined for destructive thickness tests, were also given a test for adhesion. If trouble was suspected, plating was discontinued until the cleaning cycle had been corrected. Actually, only 4 panels of the 80 tested in this way gave any indication of trouble and this was quickly corrected.

Plating of Test Specimens

One primary consideration in any test program of this nature is, of course, plate thickness. It must be uniform from point to point on a panel, from panel to panel on a plating rack, from rack to rack within a lot; and each lot must have the specified thickness. In effect, this makes the use of the Haring cell arrangement mandatory, but since a program of this size involves the plating of several thousand panels, a multipanel rack must be used, and it is only possible to approximate as closely as possible the true Haring cell. This was done by using a rack (Fig. 2) designed so that the panel faces and rack members were in the same plane and very closely spaced. The rack was left bare, to act as a thief, and was stripped regularly, to avoid undue build up. The arrangement of the rack in the tank was also very closely controlled (Fig. 3). All dimensions were carefully chosen and the position of anodes and rack were fixed by suitable stops and guides. The solution level was only ¼ in. over the upper horizontal rack member; the rack extended to within ⅝ in. of the tank wall (or a glass shield extending the length of the tank, in steel or lead-lined tanks) and the tank bottom under the rack position was built up with bricks to within ½ in. of the bottom of the rack. After the panels had been bolted to the rack (in a position dictated by the panel number) they were carefully aligned with their faces coplanar with the rack members and adjusted to give an equal ¼-in. clearance all around, between panels and rack members, and between adjacent panels. Preliminary work had shown a small but detectable difference in thickness if a panel were racked ⅛ in. or more out of line with the plane. Since this much misalignment was easily detectable in racking, it was clear that significant variations from this source were not to be expected, providing normal care was used in mounting the panels. Quartz electric-immersion heaters were used, under thermostatic control, to maintain the bath temperature. A small propeller stirrer, mounted behind the anodes, served to equalize temperature and concentration throughout the tank, without causing any significant agitation as such. Agitation was felt to be undesirable, since it is a factor that cannot be uniformly or precisely controlled, and has an effect on concentration polarization at the cathode which can increase efficiency and hence influence plating thickness. In fact, in early calibration runs, the stirrer was placed in the very corner of the tank at position *Y* (Fig. 3). It was found that the upper left-hand panel on the rack consistently showed thicker deposits than the rack average, especially in copper plating. This effect disappeared when the stirrer was moved over behind an anode (position *X*).

Each plating tank was provided with a calibrated ammeter to ensure constancy of current density and with a calibrated ampere-hour meter actuating a buzzer and signal light. A great deal of careful work was done to determine the ampere-hours required in each solution to give the desired thickness. In the calibration runs, thickness was measured on nine selected locations for each panel on the rack. The measurements were made first with the Magne-Gage, then on ½-in. square slugs punched from the selected spots. The deposits were dissolved from the face side chemically and analyzed to determine the weight of copper and nickel. Microscopic measurements were also made on sections of the panel. The chemical method was judged to be most accurate and was used to calibrate the Magne-Gage. Chromium thicknesses were measured with the hydrochloric acid spot test at five selected points.

It was recognized that in the operation of the Magne-Gage the skill of the operator is a considerable factor in obtaining accurate, reproducible measurements; also that two operators with equal ability to obtain reproducible results will not necessarily obtain the same readings on a given coating. In other words, for best results, every operator should prepare his own calibration curve for the instrument. This is especially important when two or more operators must use the Magne-Gage, as when panels are prepared by more than one source. In this program, one operator took all the measurements; the calibration curve, as stated, was prepared from a set of uniformly plated panels of varying thicknesses, which were then measured by the chemical method.

TABLE I.—EFFECT OF MAGNETISM ON MAGNE-GAGE READINGS.

Panel	A		B		C			
Magnetism:								
Compass deflection, deg	45	0	45	0	45	45	15	0
Distance from compass, in.	5	1	4	1	6	2¼	1	1
Readings on scale:[a]								
Top of panel	110	130	133	130	106	92	93	91
Middle of panel	134	130	124	124	90	93	93	93
Bottom of panel	141	129	115	122	72	87	85	86
Average reading	127	130	124	125	90	90	90	90
Standard deviation of scale readings	16.4	0.6	9	5	14.4	3.0	3.9	3.0
Corresponding standard deviation in thickness, mils	0.18	0.007	0.1	0.06	0.16	0.03	0.04	0.03

[a] Average of four readings at each level.

In calibrating the Magne-Gage, it was found (19) that, especially with one of the lots of steel (SAE 1095), there was a considerable degree of magnetism, either inherent or induced by the cleaning and plating operations. On such panels, the Magne-Gage readings were invariably high on one end of the panel and low on the other, although the average was usually quite consistent with the actual thickness as determined microscopically or chemically. Such panels gave a marked deflection to a sensitive compass, one end of the panel attracting and the other repelling the needle. A typical magnetized panel, measured in ten places, gave an average Magne-Gage reading (Table I) of 127, with a standard deviation of 16.4. When demagnetized, the average reading was 130, with a standard deviation of 0.63. It was found additionally by demagnetizing a panel in steps, that if the panel would deflect the compass needle 45 deg when held along the E-W line at a distance of 6 in. from the needle, a scatter of ±25 per cent could be expected in the readings. Demagnetizing a panel to a degree such that the needle was deflected 45 deg or less at a distance of 2¼ in. gave readings with a scatter no greater than with the fully demagnetized panel. It was concluded that partial demagnetizing (which is much less tedious than complete demagnetizing) to a controlled degree would give satisfactory control readings.

For the production runs, the Magne-Gage was used as a rough control check on two panels from each lot, after demagnetizing the panels sufficiently, as above. The use of the Magne-Gage was restricted to copper deposits 0.5 mil thick or less and to nickel deposits not over 1.5 mils. The chemical method was used for thicker deposits and the chemical and microscopic methods for composite copper-nickel deposits. Two panels from each lot of 48 were punched in two places, and the thickness of copper and nickel on the slugs was measured microscopically, front and back. An average of four measurements was made on each slug face. On the same panels, chromium thickness was measured in two places by the spot test (20). The summary of these measurements appears in Table III, together with their calculated standard deviations. It is considered that the degree of control achieved was quite good, especially when it is remembered that half of the copper-plated panels and nearly all the nickel-plated panels had been buffed, and some of the panels had two layers of buffed deposits.

TABLE II.—ANALYSIS OF RANDOM THICKNESS MEASUREMENTS FROM PROGRAM NO. 4.

	Lot A	Lot B	Lot C
Intended thickness, mils-nickel	1.45	1.5	1.5
Buffed	no	no	yes
Actual average thickness, after buffing	1.80	1.63	1.81
Standard deviation	0.30	0.26	0.05
Variation of average from intended thickness, per cent	21.0	9.0	21.0

TABLE III.—AVERAGE THICKNESS OF DEPOSITS.

Intended thickness, mils	0.5	0.9	1.0	1.4	1.5	1.9	2	3
Actual average copper thickness, mils	0.528	...	1.01	...	1.52	...	...	...
Number of determinations	40	...	60	...	24	...	...	...
Standard deviation, mils	0.082	...	0.073	...	0.1	...	...	...
Actual average nickel thickness after buffing, mils	0.531	0.981	0.994	1.431	1.462	1.875	1.975	2.99
Number of determinations	48	16	68	16	32	36	16	8
Standard deviation, mils	0.056	0.09	0.057	0.066	0.071	0.1	0.061	0.1
Standard deviation, per cent of average nickel thickness	10.5	9.2	5.8	4.6	2.2	5.3	3.1	3.3
Variation of nickel average from intended thickness, per cent	6.2	9.0	0.6	2.2	2.5	1.0	1.0	0.3

By comparison, analysis of thickness measurements of several lots selected at random from the 1956 atmospheric corrosion test Program No. 4, currently in progress, are shown in Table II.

It has sometimes been suggested that one way to conduct corrosion tests is to exercise somewhat less care in the preparation of the panels and to treat the results statistically. This is arrant nonsense, since no statistical method in the world can extract meaningful results from data that are statistically out of control.

It will be noted that the average thicknesses obtained in the 1947 Program No. 1 (Table III), were quite close to the intended thicknesses and that the standard deviations were 5 per cent or less of the average thickness for deposits 1 mil and heavier.

STANDARDIZATION OF BUFFING LOSSES

Since the final thicknesses of copper and nickel after buffing determine the corrosion behavior, it was necessary to

TABLE IV.—COMPARISON OF HAND AND MACHINE BUFFING IN PRELIMINARY STUDIES.

	Copper		Nickel	
	Hand	Machine	Hand	Machine
Number of panels	195	54	160	72
Average weight loss, mg	583	431	217	192
Average standard deviation, $\bar{\sigma}$	[a]	46.6	[a]	20.8
Average deviation[b]	87	43.6	41.5	16
Extreme deviation	+417 −263	+184 −131	+184 −100	+57 −38

[a] Original data are not available for this calculation.

[b] Average magnitude of the individual deviations from the grand average. It is shown, together with the extreme deviations, because the average standard deviation, $\bar{\sigma}$, of some of the lots, could not be calculated.

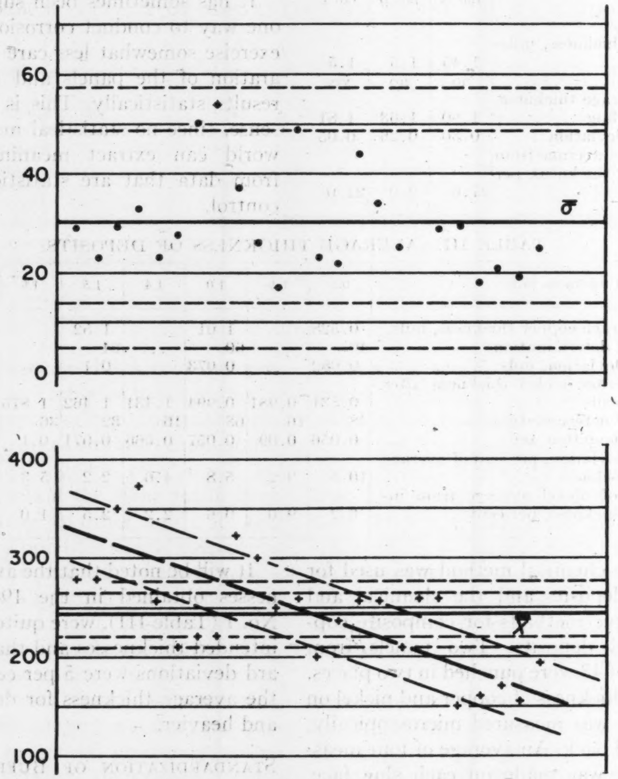


FIG. 4.—Weight Losses in Nickel Buffing in Production Runs.

Lower graph.—Successive lot averages of 29 lots of 6 panels each, average loss, mg per 4 by 6-in. panel.

Upper graph.—Standard deviation of each lot, plotted around $\bar{\sigma}$, the standard deviation of the grand lot.

standardize the buffing procedure so that, in plating, a predetermined allowance could be made for the metal to be removed in buffing. Hand-buffing was studied first, using an operator of long experience who was unusually careful and cooperative. The panels were held to the wheel by a paddle over a fulcrum, in a fashion similar to that used successfully in polishing against a belt. It was found, however, that even with the utmost care and attention, buffing losses were widely scattered, and uniform removal could not be attained (Table IV).

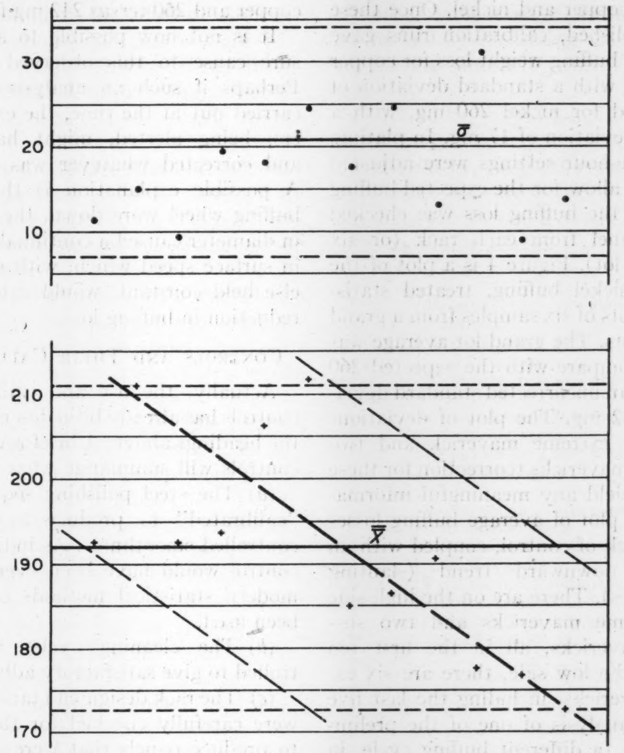


FIG. 5.—Weight Losses in Nickel Buffing in a Preliminary Calibration Run, with a Different Buffing Cycle from that of Fig. 4.

Lower graph.—Successive lot averages of 12 lots of 6 panels each, average loss, mg per 4 by 6-in. panel.

Upper graph.—Standard deviation of each lot, plotted around $\bar{\sigma}$, the standard deviation of the grand lot.

A buffing machine was then designed and built, as has been described elsewhere (21). Briefly the machine consisted of a horizontal rotating table capable of holding eight 4 by 6-in. panels in a horizontal position. The table rotated under a buffing wheel and could be raised and lowered to make or break contact with the wheel. A weighted lever arm applied a force of 70 lb against the wheel. The speed of wheel and table and the pressure were maintained constant. Buffing composition was applied by hand at regular intervals, in small amounts in as controlled a manner as possible. Two of the eight panel positions were used as dummies and each cycle was started and stopped on these dummy panels. Considerable work was done to establish the optimum cycles for copper and nickel. Once these were established, calibration runs gave an average buffing weight loss for copper of 467 mg, with a standard deviation of 30 mg, and for nickel 260 mg, with a standard deviation of 17 mg. In plating, the ampere-hour settings were adjusted upward to allow for the expected buffing loss. Also, the buffing loss was checked on one panel from each rack (or six panels per lot). Figure 4 is a plot of the data for nickel buffing, treated statistically as lots of six samples from a grand lot of 29 lots. The grand lot average was 242 mg (compare with the expected 260 mg), with an uncorrected standard deviation of 31.2 mg. The plot of deviations shows one extreme maverick and two suspected mavericks (correction for these does not yield any meaningful information). The plot of average buffing losses shows a lack of control, coupled with an apparent downward trend (slanting dashed lines). There are on the high side four extreme mavericks and two suspected mavericks, all in the first ten runs. On the low side, there are six extreme mavericks, including the last five runs. An analysis of one of the preliminary tests (a different buffing cycle, in which 72 determinations were made, in 12 lots of 6) shows an exactly similar behavior (Fig. 5). In this case there was only one suspected maverick in the average losses and none in the standard deviations. It is perhaps interesting that if one draws the best line through the points of Fig. 5 and applies the same limits to the new line of changing averages (slanting dashed lines) there are no suspected mavericks, a strong indication that the downward trend is real. The data on copper buffing were not numerous enough to show any trend, if it existed. However, the calibration runs cited above also gave higher average losses for both copper and nickel than the later production runs (467 *versus* 431 mg for copper and 260 *versus* 242 mg for nickel).

It is not now possible to assign any sure cause to this observed behavior. Perhaps if such an analysis had been carried out at the time, the experimenters, being alerted, might have found and corrected whatever was occurring. A possible explanation is that as the buffing wheel wore down, the reduction in diameter caused a continual reduction in surface speed which, with everything else held constant, would cause such a reduction in buffing loss.

CONTROLS AND THEIR CALIBRATION

Actually, the use and calibration of controls has already been described under the headings above. A brief review of the controls will summarize what was done:

(*a*) The steel polishing sequence was "calibrated" to produce a surface of controlled smoothness. As indicated, the control would have been even better if modern statistical methods could have been used.

(*b*) The cleaning cycles were controlled to give satisfactory adhesion.

(*c*) The rack design and tank geometry were carefully checked for their ability to produce panels that were of uniform thickness regardless of the position on the rack.

(*d*) The geometry of the plating set-up was controlled to give maximum uniformity of deposit.

(*e*) The usual chemical and instrumental controls over plating solution and conditions were used.

(*f*) Ampere-hour meters were used to control plating thickness. They were calibrated by measuring actual thickness obtained in the particular geometry of the set-up used.

(*g*) Buffing was done mechanically in a reproducible way, controlled by measuring the weight loss. Allowance was made in plating time to compensate for average buffing losses.

(*h*) To eliminate the effect of obvious chance variations, all panels were inspected for visible defects arising from steel imperfections, mishandling or other accidental causes. These panels were discarded.

(*i*) To eliminate the effect of systematic variation from panel to panel on a plating rack, randomization was used, as described below, on the remaining satisfactory panels.

(*j*) Boxes were made with individual compartments for carrying and temporary storing of panels in order to avoid injury to the surface finish. These were used throughout the program.

By random number tables, the panels were randomized into 9 groups of 5 each. No two panels in any group were from the same plating rack. Panels Nos. 1 and 31 were used for thickness and adhesion tests, as described, while the remaining panel No. 43 was held as a possible substitute to replace a panel showing some obvious defect after plating. Since there were six exposure sites and one salt spray test planned, it was only necessary to choose seven groups at random for these tests, leaving two unused groups for further substitute panels, if required.

An indication of the uniformity of the samples may be had from the salt spray data on 38 lots of 5 samples each, all tested in the same box at the same time at Chrysler Engineering Laboratories (22). The number of rust spots at 500 hr was counted for each panel and averaged for each lot. It was found that the individual deviation from the average was within the expected Poisson distribution in 29 of the lots, for probability limits 0.1 and 0.9, and in all but one of the lots for probability limits 0.005 and 0.995. One lot, with an average number of 23.4 spots, which between the wider limits would be expected to show from 12 to 38 spots on individual panels, actually had from 10 to 44 spots.

Another indication of the uniformity may be had by examining the salt spray data for the same 190 panels in another way:

(*a*) At 96 hr, only 3 panels had failed (2 or more rust spots);

(*b*) At 96 hr, only 10 panels showed one or more rust spots; five of these were from lots that showed average lot failure in 160 to 300 hr, and

(*c*) At 500 hr:

Between narrow Poisson limits	45 panels in 9 lots showed no rust spots
	35 panels in 7 lots showed from 0 to 1 rust spot
	35 panels in 7 lots showed from 0 to 3 rust spots
	20 panels in 4 lots showed from 0 to 5 rust spots
	10 panels in 2 lots showed from 1 to 5 rust spots
Between wide Poisson limits	10 panels in 2 lots showed from 0 to 10 rust spots
	5 panels in 1 lot showed from 0 to 15 rust spots
	5 panels in 1 lot showed from 0 to 20 rust spots
	20 panels in 4 lots showed from 2 to 15 rust spots
	5 panels in 1 lot showed from 10 to 44 rust spots

While one may quarrel with the validity of the salt spray test as a yardstick of actual performance, it can be a carefully controlled one-shot corrosion test and as such is also a test of the uniformity of samples within a lot, especially when all are tested in the same box at the same time.

Also, 38 similar lots were exposed at each of the six atmospheric test locations. Unfortunately, it was not until the 1956 Program No. 4 that atmospheric exposure panels were given individual ratings. Consequently, no statistical appraisal of uniformity is possible for the 1947 Program No. 1, but still the data could be consistently interpreted and were, within expected differences due to varying severity of atmosphere, consistent from site to site. Pictures of some of the Kure Beach, N. C. exposure panels have been published (14) which well illustrate the uniformity of attack.

DISCUSSION

Public acceptance of plated products, particularly of automobile hardware, is the life-blood of the plating industry. This acceptance must in the long run be based upon performance of the coatings in service. Specifications for plated coatings are issued for the purpose of offering products that will meet with public approval at the lowest possible cost. If they are to have meaning, these specifications must be written in the light of results obtained in exposure tests. The interpretation of such results must not be clouded by any doubt that the test panels really are what the schedule of the test program says they should be.

The only way to be sure of obtaining truly representative panels is first to recognize that their preparation is not a production job but one of research, in spite of the large number of panels to be prepared. No one would think of plating test panels on a fully automatic plating machine unless the purpose were to compare this machine with another one or with manual operations. It is just as futile to attempt their preparation in a manual production tank, even in a quiet corner of the shop with the program supervisor sitting only a few yards away. Everything from the polishing of the basis metal to the buffing of the final plate must be done under laboratory conditions. Each piece of equipment used must be tailored to the job, in a recognized standard fashion. Methods which are recognized to contribute to uniformity of performance should be adopted as standard. As a specific instance, a standard plating rack should be used in a standard tank arrangement approaching the Haring cell design and equipped with specified automatic control means, including calibrated ampere-hour meter recording ammeter and temperature controls.

Detailed and complete records must be kept of every variable that can be measured. Statistical quality control methods should be used to randomize sampling and to reveal and check deviations from standard limits before they occur. To make this possible, the program must be designed and laid out so that valid statistics can be obtained. Calibration runs should be made to establish quality control standards for every operation that can be thus controlled. In these runs, it will be possible to detect other than chance variations and to correlate them with the operating procedure, so that in the production runs corrective steps can be taken when the control charts indicate a correction is necessary. The results of all these statistical analyses should be included in the summary report on the results of the exposure test, so that it will be possible to judge the efficacy of the controls.

In the 1956 Program No. 4, a welcome innovation in inspection procedure was made in that each panel was individually rated. In future programs, then, it will be possible to exert statistical control over all phases of panel preparation and also to examine the results for statistical significance of the variables under study, with the resulting assurance that any conclusions which are suggested may actually be tested for validity and issued with confidence.

REFERENCES

(1) W. A. Wesley, "Rating Exposure Test Panels of Decorative Electrodeposited Cathodic Coatings," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 803 (1947).

(2) H. A. Pray, "Inspection of Exposure Test Panels with Nondecorative Electrodeposited Cathodic Coatings," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 822 (1947).

(3) K. G. Soderberg, "Inspection of Exposure Test Panels with Anodic Electrodeposited Coatings," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 826 (1947).

(4) W. Blum, P. W. C. Strausser, and A. Brenner, "Protective Value of Nickel and Chromium Plating on Steel," *Journal of Research*, National Bureau of Standards, Vol. 13, No. 3, p. 331 (1934).

(5) W. Blum, P. W. C. Strausser, and A. Brenner, "Corrosion-Protective Value of Electrodeposited Zinc and Cadmium Coatings on Steel," *Journal of Research*, National Bureau of Standards, Vol. 16, No. 2, p. 185 (1936).

(6) W. Blum and P. W. C. Strausser, "Outdoor Exposure Tests of Electrodeposited Nickel and Chromium Coatings on Steel and Nonferrous Metals," *Journal of Research*, National Bureau of Standards, Vol. 24, No. 4, p. 443 (1940).

(7) C. H. Sample, "Inspection and Rating of Test Panels Employed in Outdoor Exposure Tests of Electroplated Coatings," *Monthly Review*, Am. Electroplaters' Soc., Vol. 32, p. 23 (1945).

(8) P. W. C. Strausser, "Progress Report on Exposure Tests of Plated Coatings," *Monthly Review*, Am. Electroplaters' Soc., Vol. 19, No. 2, p. 15 (1932).

(9) W. A. Blum, "Report on the Preparation of Specimens Used for Exposure Tests of Electrodeposited Metal Coatings on Steel," *Monthly Review*, Am. Electroplaters' Soc., Vol. 19, No. 5, p. 6 (1932).

(10) W. A. Blum, "Protective Value of Electroplated Metal Coatings on Steel," *Monthly Review*, Am. Electroplaters' Soc., Vol. 19, No. 9, p. 3 (1933).

(11) W. A. Blum, "Protective Value of Electroplated Metal Coatings on Steel," *Monthly Review*, Am. Electroplaters' Soc., Vol. 19, No. 10, p. 37 (1933).

(12) P. W. C. Strausser, "Experiences in the Plating of Samples for Exposure Tests," *Monthly Review*, Am. Electroplaters' Soc., Vol. 19, No. 10, p. 40 (1933).

(13) M. R. Caldwell, L. B. Sperry, L. M. Morse, and H. K. DeLong, "Corrosion Resistance of Copper-Nickel-Chromium Plated Zinc-, Aluminum-, and Magnesium-Base Die Castings," *Plating*, Am. Electroplaters' Soc., Vol. 39, pp. 142, 159 (1952).

(14) Report of Subcommittee II on Performance Tests of ASTM Committee B-8 on Electrodeposited Coatings, *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 226 (1949).

(15) Report of Subcommittee II on Performance Tests of ASTM Committee B-8 on Electrodeposited Coatings, *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 204 (1951).

(16) Report of Subcommittee II on Performance Tests of ASTM Committee B-8 on Electrodeposited Coatings, *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 256 (1953).

(17) Report of Subcommittee II on Performance Tests of ASTM Committee B-8 on Electrodeposited Coatings, *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 331 (1955).

(18) A. K. Graham, "Notes on Cleaning Cycles for Plating," *Transactions*, Fourth Internat. Conference, Inst. of Metal Finishers, Vol. 31, p. 259 (1954).

(19) G. B. Bowman, "Magnetism and Magnetic Thickness Gage," *Monthly Review*, Am. Electroplaters' Soc., Vol. 33, pp. 1044, 1092 (1946).

(20) Methods of Test for Local Thickness of Electrodeposited Coatings, (A 219 – 58), 1958 Book of ASTM Standards, Part 3.

(21) C. C. Cupps and A. K. Graham, "Standardization of Buffing for Preparation of Atmospheric Exposure Test Panels," *Proceedings*, Am. Electroplaters' Soc., Vol. 29, p. 19 (1942).

(22) C. H. Sample, "The Corrosion Behavior and Protective Value of Copper-Nickel-Chromium and Nickel-Chromium Coatings on Steel," Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings, Am. Soc. Testing Mats., p. 7 (1956). (Issued as separate technical publication *ASTM STP No. 197*.)

REPORT OF COMMITTEE B-9
ON
METAL POWDERS AND METAL POWDER PRODUCTS*

Committee B-9 on Metal Powders and Metal Powder Products held one meeting during the year: in Pittsburgh, Pa., on February 3, 1959.

NEW TENTATIVES

The committee recommends for publication as tentative the following method and specifications as appended hereto:[1]

Tentative Method for:

Hardness Testing of Sintered Metal Friction Materials.

Tentative Specifications for:

Machinable High-Density Tungsten-Nickel-Copper Alloys.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of:

Subsieve Analysis of Granular Metal Powders by Air Classification (**B 293 – 54 T**).

Tentative Recommended Practice for:

Hardness Testing of Cemented Carbides (**B 294 – 54 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ADVISORY COMMITTEE ACTIVITIES

The Advisory Committee held two meetings during the year. A number of committee personnel changes were approved. Mr. W. W. Wellborn was appointed Chairman of Subcommittee III-C on Cemented Carbides upon the resignation of Mr. Engle. An Award of Merit Committee consisting of Mr. Koehring, chairman, H. R. Biehl, and K. H. Roll was appointed. The committee reviewed the progress being made in publishing the Committee B-9 specifications and test methods in a separate volume. A special introduction to this volume is being prepared.

ACTIVITIES OF SUBCOMMITTEES

The eight active subcommittees and sections of the committee met twice during the year: in Cleveland, Ohio, on October, 1958, and prior to the main committee meeting in Pittsburgh, Pa., on February 2 and 3, 1959.

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman) is continuing its research activities in the area of gathering data on density *versus* tensile strength properties of sintering iron powder with 0.5 per cent zinc stearate. Due to extreme variations in the data reported by cooperating laboratories no standard correlation can be established. Study of data revealed the fact that all data submitted gave a family of curves of approximately the same slope. From this it was concluded that fewer density levels could be investigated in subsequent tests without affecting the validity of the curves. Under the leadership of Mr. Geijer a closely controlled program is being carried out between cooperating laboratories. The tension test bar design is being reviewed to determine if a design change might be advisable. Still under consideration is a name for the end product of the powder metallurgy industry.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 2.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Metal Powders (J. B. Haertlein, chairman):

Section A on Base Metal Powders (J. B. Haertlein, chairman) has finished its work on methods of testing the physical properties of metal powders and is now testing the mechanical properties obtained when using various powders so that ultimately specifications can be drawn up for the powders themselves. A task group headed by F. I. Zaleski has drawn up tentative plans for a sintering furnace. Another group headed by P. V. Schneider has conducted a cooperative testing program on sintering copper-tin which has indicated the poor reproducibility now obtainable in the industry. This work is all of a research nature and will require extensive and careful contributions from all participating individuals and laboratories. The subcommittee is also considering a revised thief for sampling metal powders and a new specification for compressibility of iron in the high pressure range (50 tons per sq in. and above).

Section B on Refractory Metal Powders (W. H. Bleecker, chairman) is cooperating with Committee E-3 in conducting research on chemical analysis. A round-robin test demonstrated a serious error in the hydrogen weight loss test, and work is continuing to determine the cause of this error. The Task Group on Turbidimetric Test Method has prepared three different methods of testing, all using the Cenco photelometer. These methods will be investigated by four laboratories who will report on each of the three test procedures using fractions of the same tungsten powder.

The task group working in the field of the microscopic test method has not been able to obtain good reproducibility from one laboratory to the other.

Subcommittee III on Metal Powder Products (R. P. Koehring, chairman):

Section A on Bearings (M. H. Meighan, chairman) prepared a tentative specification for flange and thrust washer diameters which is being reviewed by the Editorial Subcommittee. A task group is working on a specification for the lubricant to be recommended for use as an impregnating lubricant in metal powder sintered bearings.

The section voted to revise the Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 – 58 T) by changing the restrictive limits on type II, class B bearings represented by the 5.0 to 30 per cent copper composition. The task group working on the correlation of K-factor, microstructure, and machinability has made a progress report. This group has done a tremendous amount of research and has accumulated a great deal of data from which a paper will be prepared for publication by the Society. The section voted to raise the K-factor for type I class A and class B bearings from 22,500 to a value of 26,500 in Specifications B 202.

Section B on Structural Parts (P. J. Shipe, chairman) has approved the correction factors for the hardness testing of round specimens as submitted by Subcommittee 6 of Committee E-I. A task group is working on the problem of finding a suitable method for measuring the surface finish of a porous metal part. The section prepared the preface to the publication of Committee B-9 specifications and test methods in a separate volume. The section has approved the suggestion for a specification applicable to iron-copper-carbon sintered parts. Also, a task group of this section is drafting an additional specification for high density iron.

Section C on Cemented Carbides (W. W. Wellborn, chairman) is working on a revision of the Recommended Practice for Hardness Testing of Cemented Carbides (B 294 – 54 T). This section is working closely with the Cemented Carbide Producers Assn. to bring about a better standardized testing procedure and is now working on a new approach in which it will bring within the current testing procedures those tests that are now applicable to cemented carbides.

Section D on Friction Materials (H. B. Huntress, chairman) drafted the proposed Tentative Method for Hardness Testing of Sintered Metal Friction Materials referred to earlier in the report. The committee voted to change the name and scope of the material under consideration from "friction material" to "sintered metal friction material." A test method for density has been prepared and is being submitted for approval of this section. Cooperative tests are being conducted using a proposed method for a transverse rupture test in an effort to determine the effect of loading rate on a variety of sintered metal friction materials. Research into the areas of thermal properties and friction testing continues.

Section E on Machinable Heavy Metals (A. S. Doty, chairman) prepared the proposed Tentative Specification for Machinable High-Density Tungsten-Nickel-Copper Alloys referred to earlier in the report. This section recommended that it become inactive after the completion and publication by the Society of this proposed tentative.

This report has been submitted to a letter ballot of the committee, which consists of 79 members; 58 members returned their ballots, of whom 54 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. L. BONANNO,
Chairman.

C. G. JOHNSON,
Secretary.

REPORT OF ADVISORY COMMITTEE
ON
CORROSION

The Advisory Committee on Corrosion held two meetings during the year: on June 24, 1958, in Boston, Mass., and on November 20, 1958, at ASTM Headquarters, Philadelphia, Pa.

PERSONNEL

New committee representatives have been appointed for Committee C-19 as follows: T. P. Pajak replaces George Gerard, and L. E. Johnson replaces J. H. Gibbud.

O. B. Ellis and W. H. Finkeldey were recommended for appointment as members-at-large for three-year terms beginning June, 1959.

The officers elected for the ensuing term of two years are as follows:

Chairman, K. G. Compton.
First Vice-Chairman, C. P. Larrabee.
Second Vice-Chairman, C. H. Sample.

STATUS OF ATMOSPHERIC EXPOSURE PROGRAMS

Committee A-5 on Corrosion of Iron and Steel:

A series of approximately 750 hardware specimens using a variety of aluminum and zinc coatings on carbon steel, low-alloy steel, and nodular iron base metals are being exposed at Newark, N. J. (New York Area), and at the 80- and 800-ft sites at Kure Beach, N. C. The specimens were placed on exposure in June, 1958, to determine time to coating failure and will be removed for pit depth measurements over a period of 20 years (11).[1] This atmospheric exposure test program is more comprehensive than the first hardware program initiated in 1929 (2).

A new exposure program is being developed to compare differences, if any, between the atmospheric corrosion resistance of individually dipped as against continuously dipped roofing sheet. Aluminum-coated sheet will also be included.

A new atmospheric exposure program has been authorized to test aluminum-coated wire for farm field fencing, chain link fencing, barbed and plain wire strand.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

A total of 4184 specimens of 17 grades of stainless steels were placed on exposure during 1958 at the New York Area (Newark, N. J.); Columbus, Ohio; State College, Pa.; Freeport, Tex.; and the 80- and 800-ft sites at Kure Beach, N. C. The specimens include spot-welded panels, arc welded panels, tension panels, Erickson cup panels, 0.505-in. tension test bar specimens, panels for evaluation of appearance, and static and loaded coil springs for determination of fatigue properties. These properties will be determined for atmospheric exposures of 3, 7, and 15 yr.

---

[1] The boldface numbers in parentheses refer to the list of references appended to this report.

Committee B-3 on Corrosion of Non-Ferrous Metals:

Subcommittee VI on Atmospheric Corrosion.—Twenty-two alloys of aluminum, some in several heat treatments, and some clad aluminum, totaling 34 groups of specimens, were exposed at Kure Beach, N. C. (80-ft site), New York Area (Newark, N. J.), Point Reyes, Calif., and State College, Pa., for 1, 2, 7, and 20 years. There are also a total of 42 other metals and alloys including copper, lead, magnesium, molybdenum, nickel, titanium, and zinc exposed at the same locations for 2-, 7-, and 20-yr periods. Tantalum specimens were exposed for these three time periods at Kure Beach and Newark only. The degree of corrosion will be determined by weight, tensile strength, and pit depth measurements. A preliminary report of the initial data will appear in the 1959 *Proceedings*.[2]

Subcommittee VII on Weather.—A report on the calibration of the relative corrosivity of ASTM test sites will appear in the 1959 *Proceedings*.[3]

The final report on the measurement of surface moisture as related to the corrosion rate of steel was presented. A sensing element has been developed for detection of surface moisture. These have been installed at several test sites. Correlations of these data with sulfur dioxide measurements were made.

Subcommittee VIII on Galvanic and Electrolytic Corrosion.—Two magnesium alloys were exposed in contact with aluminum alloys, mild steel, red brass, 18-8 stainless steel, chromium plated steel, and monel metal for periods of 5, 10, and 15 years at New York Area (Newark, N. J.), State College, Pa., and Kure Beach, N. C. (80- and 800-ft sites). There were 165 panel-type galvanic couples exposed at each test site. The degree of corrosion will be determined by tension tests on specimens cut from the magnesium panels. Visual inspection and measurements of pit depth will also be recorded.

The stainless-steel couples removed from atmospheric exposure sites after 15 years of exposure are being tested in preparation for the final report of the program begun in 1941.

Committee B-6 on Die-Cast Metals and Alloys:

The 20-yr exposure data on the 1935 exposure test evaluating die-casting alloys (6) was circulated within the committee in 1955 for review. A report correlating all of these data from 20-yr exposure will be appended to the 1960 Annual Report of Committee B-6 (6).

All remaining bars from the 1939 exposure test evaluating zinc alloys 21, 23, and 25 and magnesium alloys 307, 312, 313, and 314 have been collected and are being tested for the 20-yr data.

The 6-yr specimens from the 1953 exposure test program evaluating SC84A aluminum die-casting alloys (7) have been collected and testing is complete.

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Twenty-seven aluminum and eight magnesium alloys totaling over 5800 specimens were exposed during 1951 to 1954 at State College, Pa.; New York Area (Port Authority Building, N. Y.); the 800-ft site at Kure Beach, N. C.; Point Reyes, Calif.; and Freeport, Tex.

The specimens include riveted and spot-gas and arc-welded wrought and clad panels, and sand- and permanent-mold cast tension bars. The specimens are being measured to obtain data on change in tensile strength, yield strength, and elongation over exposure periods of 1, 3, 5, and 10 yr. The 1- and 3-yr reports for these exposures have been published (10). The 5-yr specimens have been withdrawn for evaluation.

---

[2] See p. 176.

[3] See p. 183.

Anodic coated panels were also included for visual observations only.

Committee B-8 on Electrodeposited Metallic Coatings:

A program to evaluate decorative electroplated coatings of copper, nickel, and chromium on aluminum alloys is in preparation. The objectives are: (1) to compare the corrosion resistance offered by nickel plating to several aluminum alloys; (2) to compare Watts nickel with bright nickel for this application; (3) to compare the corrosion performance of different base metals with similar deposits; and (4) to determine the effect of heavy copper layers under nickel.

TABLE I.—CORROSION TESTS OF ALUMINUM AND ITS ALLOYS PUBLISHED BY ASTM.

The boldface numbers refer to the list of references appended to this report.

Aluminum	Industrial[a] Atmosphere	Marine[a] Atmosphere	Rural[a] Atmosphere	Desert[a] Atmosphere	Indoor Exposure	Salt Spray	Liquid Immersion
Wrought (Rolled, Drawn)..	**4, 10, 15, 16, 17, 18, 19**	**4, 10, 15, 16, 17, 19**	**4, 10**	**4**	...	**1, 18, 21**	**1, 15, 17, 20**
Pressure Die-Cast..........	**3, 6, 7, 8**	**3, 6, 7, 8**	**3**	...	**3, 6**	**6, 8, 19**	**6, 19**
Permanent Mold Castings.	**10**	**10**	**10**				
Sand Mold Castings.......	**10**	**10**	**10**	...	...	...	...
Hot-Dipped Coatings......	**2, 12**	**2, 12**	**2**	...	...	...	...
Clad Coatings............	**16, 19**	**16, 19**	...	...	...	...	...
Sprayed Coatings (Metalized)...................	**9, 11**	**9, 11**	**9**	...	...	...	**9**
Galvanic Cell............	**5, 12, 13, 14**	**5, 12, 13, 14**	**5, 12, 13, 14**	**5**	...	...	**16**

[a] Details concerning test sites currently maintained by ASTM of this type of atmosphere appear in the *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 229 (1958).

Committee C-19 on Structural Sandwich Constructions:

In 1955 a series of sandwich panels having facings of aluminum (some anodized), fiber glass, plate glass, kraft paper, stainless steel, and porcelain enameled steel with cores of aluminum, kraft paper, and balsa were placed on exposure at State College, Pa., and the 80-ft site at Kure Beach, N. C. These specimens were withdrawn in 1956 when a similar series was exposed for periods of 1, 2, and 3 yr. A third test program was exposed in April, 1959, and includes 9 types of honeycomb sandwich constructions. Panel faces included plain and porcelain enameled aluminum alloys, hot-dip zinc on steel, glass fiber laminates, and Formica. Cores are various weights of phenolic-impregnated paperboard with a variety of adhesives.

American Welding Society:

A large program was set up in 1953 to amass atmospheric exposure data on metalized (sprayed) coatings. Twenty-eight varieties each of aluminum and zinc coatings on steel were exposed. Half of these specimens have additional organic coatings in both single and double coatings since the porosity of metalized coatings is a limiting factor to their usefulness. Exposures, which have recently been extended, now include 1, 3, 12, and 18 yr at the 80- and 800-ft sites at Kure Beach, N. C.; Newark, N. J. (New York Area); Columbus, Ohio; East Chicago, Ind.; Freeport, Tex.; Point Reyes, Calif.; and three sea water exposures. The results following 3 to 3½ yr exposure have been published (9).

ALUMINUM CORROSION TESTING

The corrosion resistance of aluminum and its alloys has played a leading role in its expanding utilization since the first commercial appearance in 1888. About 1922, ASTM Committees A-5 and B-2 began to formulate corrosion test studies of the metal and in 1928 Committee B-7 on Light Metals and Alloys, Cast and Wrought, was established to formulate standards for this metal and its alloys. Since that time Committees B-3, B-6, B-7, and B-8 have included aluminum in atmospheric exposure test programs.

Studies under the auspices of ASTM have included outdoor and indoor exposures in various locations in North America, and salt-spray and steam-chamber tests, and sea water immersion. Various forms of aluminum and its alloys have included wrought metal and castings, hot-dipped coatings, sprayed (metalized), and clad metal, as well as aluminum coupled with other metals to form galvanic cells.

Table I lists the aluminum corrosion studies which have appeared in ASTM literature and those programs under the sponsorship of the Society's technical committees; a list of references for these programs is given.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

S. F. ETRIS,
Secretary.

REFERENCES

(1) Commercial grades of aluminum, admiralty metal, copper, lead, nickel, and zinc for total immersion, alternate immersion, and spray testing (using solutions of acids, bases, ammonia, and bichromate), and electrolytic immersion tests. Results are published in the *Proceedings*, Am. Soc. Testing Mats., as follows:

Program information: Vol. 24, I, p. 281 (1924).
Preparation of specimens and methods: Vol. 25, I, p. 150 (1925).
Results of tests: Vol. 27, I p. 281 (1927).

(2) Aluminum and other coatings on hardware were exposed in the atmosphere beginning December, 1928. All tests are now completed except that at State College, Pa., which is to continue indefinitely. Results of these exposures are published in the *Proceedings*, Am. Soc. Testing Mats., as follows:

Program information: Vol. 25, I, p. 115 (1925).
Data, 1- to 2-yr exposure: Vol. 31, I, p. 184 (1931).
Data, 3- to 4-yr exposure: Vol. 33, I, p. 156 (1933).
Data, 5- to 9-yr exposure: Vol. 38, I, p. 84 (1938).
Data, 9- to 14-yr exposure: Vol. 44, p. 96 (1944).
Data, 14- to 17-yr exposure: Vol. 46, p. 158 (1946).
Data, 17- to 19-yr exposure: Vol. 48, p. 119 (1948).
Data 19- to 21-yr exposure: Vol. 50, p. 114 (1950).
Data, 21- to 23-yr exposure: Vol. 52, p. 119 (1952).
Data, 24- to 25-yr exposure: Vol. 55, p. 149 (1955).
Data, 26- to 28-yr exposure: Vol. 57, p. 130 (1957).
Data, 28- to 30-yr exposure: In preparation.

(3) Twelve aluminum-base and 9 zinc-base die-casting alloys were tested for 1-, 5-, and 15-yr periods starting in June, 1929, for change in tensile strength, elongation, and Charpy impact. Program data are published in the *Proceedings*, Am. Soc. Testing Mats., as follows:

Program and specimen drawings: Vol. 28, I, p. 202 (1928).
Chemical analysis and physical data: Vol. 29, I, p. 192 (1929).

Data, 1-yr exposure: Vol. 32, I, p. 265 (1932)
Data, 5-yr exposure; Vol. 35, I, p. 190 (1935).
Data, 10-yr exposure; Vol. 40, p. 195 (1940).
Final 15-yr data: Vol. 46, p. 244 (1946).

(4) A variety of 24 alloys including 5 aluminum alloys utilizing 9 by 12 in. plates and tension test bars were exposed in 1931 to determine change in weight, tensile strength, and ductility. Information obtained was published by ASTM as follows:

Original data: *Proceedings*, Vol. 32, I, p. 214 (1932).
Analysis of 1-yr data: *STP No. 17*, p. 69 (1934).
Data, 1- and 3-yr exposure: *Proceedings*, Vol. 35, I, p. 142 (1935).
Discussion of 10-yr data: *STP No. 67*, p. 57 (1946).
Statistical analysis of 6-yr data: *Proceedings*, Vol. 41, p. 172 (1941).
Data, 1-, 3-, 6-, 10-yr exposure: *Proceedings*, Vol. 43, p. 137 (1943).
Data covering 1-yr change in properties: *Proceedings*, Vol. 44, p. 224 (1944).
Data, 20-yr exposure: *STP No. 175*, p. 3 (1955).
Discussion of aluminum tests: *STP No. 175*, p. 21 (1955).

(5) Aluminum bolted to six other metals to form galvanic cells was exposed in 1931 at nine test sites for 7 years. Data published in *Proceedings*, Am. Soc. Testing Mats., as follows:

Data, 1-yr exposure: Vol. 34, I, p. 236 (1934).
Data, 3-yr exposure: Vol. 35, I, p. 167 (1935).
Data, 7-yr exposure: Vol. 39, p. 247 (1939).

(6) Three die-cast aluminum alloys (IVa, V, Va, Specifications B 85 – 39 T), exposed in 1934 to determine change in tensile strength and elongation. Subjected to salt spray, alternate immersion, indoor, and atmospheric exposure for 5, 10, and 20 yr. A discussion of significant information derived from this study is given in the ASTM BULLETIN, No. 163, Jan., 1950, p. 51 (TP 1). Data published in *Proceedings*, Am. Soc. Testing Mats., as follows:

Program and original data: Vol. 35, I, p. 184 (1935).
Spray and immersion tests: Vol. 36, I, p. 182 (1936).
Data, 5-yr exposure: Vol. 40, p. 191 (1940).
Data, 10-yr exposure: Vol. 46, p. 225 (1946).
Data, 20-yr exposure: In preparation.

(7) Effect of zinc content on the mechanical properties of the SC84A aluminum die-casting alloy (Specifications B 85 – 58 T). Changes in tensile strength, per cent elongation, and 0.2 per cent offset yield strength are being determined following exposures of 1, 3, 6, and 12 yr. Data published in *Proceedings*, Am. Soc. Testing Mats., as follows:

Mechanical data: Vol. 53, p. 212 (1953).
Data, 1-yr exposure: Vol. 55, p. 246 (1955).
Data, 3-yr exposure: Vol. 57, p. 174 (1957).
Data, 6-yr exposure: In preparation.

(8) Aluminum-base die-casting alloys containing 9.5 per cent silicon and 0.5 per cent magnesium or containing 8 per cent magnesium were exposed in 1944 to determine change in mechanical properties. Originally alloys G2, SG2, and SG3, (Specification B 85 – 49 T), redesignated as G8A and SG100A (Specification B 85 – 58 T). Subjected to 10-yr atmospheric exposure and 20 per cent salt spray. Data published in *Proceedings*, Am. Soc. Testing Mats., as follows:

Salt spray and atmospheric (1, 2 yr) data: Vol. 48, p. 191 (1948).
Data, 1-, 3-, and 5-yr exposure: Vol. 53, p. 207 (1953).
Data, 10-yr exposure: Vol. 58, p. 167 (1958).

(9) The American Welding Society exposed, in 1953, 28 varieties each of aluminum and zinc metalized (sprayed) coatings on steel with supplementary organic coatings. Specimens are being evaluated for pinholes, cracking, peeling, and reduction of coating thickness for periods up to 18 yr. A report covering 3 to 3½ yr of exposure is published in the *Journal*, Am. Welding Soc., Vol. 38, No. 3, p. 215 (1959).

(10) Specimens of 27 aluminum and 8 magnesium commercial alloys in the form of sheet and plate panels, welded and riveted specimens, as well as sand and permanent mold castings, were exposed in 1953 to obtain data on change of tensile strength, elongation, and yield strength. Anodic treated specimens for visual observation are included. Data published in *Proceedings*, Am. Soc. Testing Mats., as follows:

Preliminary information: Vol. 53, p. 227 (1953).
Data, 1-yr exposure: Vol. 54, p. 270 (1954); and Vol. 55, p. 284 (1955).
Data 3-yr exposure: Vol. 58, p. 194 (1958); and Vol. 59, p. 289 (1959).

(11) Hot-dipped and sprayed aluminum as well as zinc-coated hardware was exposed in

1958 to determine corrosion pit depth and time to coating failure over a 20-yr exposure period. Information concerning the program appears in *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 133 (1959).

(12) Stainless steels bolted to 3 aluminum alloys and eight other metals to form galvanic cells were exposed in 1941. Data published in *Proceedings*, Am. Soc. Testing Mats., as follows:

Specimens exposed: Vol. 42 p. 171 (1942).
Inspection 2-yr exposure: Vol. 44, p. 223 (1944).
Data, 5-yr exposure: Vol. 48, p. 167 (1948).
Removal of iron couples: Vol. 50, p. 138 (1950).
Data, 10-yr exposure: In preparation.

(13) Eighteen metals including 4 aluminum alloys were bolted to two magnesium alloys to form galvanic cells (Part I of a 3-part program). Exposures were from 2 to 4 years beginning in 1949. Symposium on Atmospheric Corrosion of Non-Ferrous Metals, Am. Soc. Testing Mats., p. 89 (1955). (Issued as separate publication *ASTM STP No. 175.*)

(14) Twenty-one metals including 2 aluminum alloys using the threaded bolt technique of forming galvanic cells were exposed in 1951 for periods from 4 months to 1 yr (Part II of a 3-part program). Complete data: *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 150 (1954). Discussion of data: Symposium on Atmospheric Corrosion of Non-Ferrous Metals, Am. Soc. Testing Mats., p. 116 (1955). (Issued as separate publication *ASTM STP No. 175.*)

(15) E. H. Dix, Jr., "Corrosion Resistance of Structural Aluminum," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 405 (1933). Atmospheric exposure and alternate immersion in 3½ per cent sea-salt solution of duralumin alloy (17ST) in a variety of shapes.

(16) F. L. LaQue and G. L. Cox, "Some Observations of the Potentials of Metals and Alloys in Sea Water," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 670 (1940).

(17) J. D. Edwards, "A Comparison of the Performance of Anodic Coatings on Wrought Aluminum Alloys When Exposed to Salt Spray and to the Weather." Salt spray data in *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 182 (1943). Atmospheric exposure data, *Ibid.*, Vol. 45, p. 146 (1945).

(18) G. R. Gohn and L. E. Menges, "Atmospheric Indoor Aging Studies on Some Aluminum and Zinc-Base Die-Casting Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1064 (1946). Discussion of 15-yr data of program in reference (7).

(19) F. M. Reinhart and G. A. Ellinger, "Effect of 20-Year Marine Atmosphere Exposure on Some Aluminum Alloys," Symposium on Atmospheric Corrosion of Non-Ferrous Metals, Am. Soc. Testing Mats., p. 47 (1955). (Issued as separate publication *ASTM STP No. 175.*) All commercially available structural aluminum alloys as well as some experimental alloys were exposed in 1932 under the auspices of the National Bureau of Standards. Heat treatment, surface treatments, and paint systems are discussed. Data are given on change of mechanical properties over atmospheric exposure periods up to 22 yr.

(20) T. J. Lummerson, M. J. Pryor, D. S. Deir, and R. J. Hogan, "Pit Depth Measurements as a Means of Evaluating the Corrosion Resistance of Aluminum in Sea Water," Papers on Metals, Am. Soc. Testing Mats., p. 157 (1957). (Issued as separate publication *ASTM STP No. 196.*)

REPORT OF THE JOINT ASTM-ASME COMMITTEE

ON

EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS*

Meetings:

Meetings of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals, its panels, and subcommittees were held during the past year in connection with the Annual Meeting of ASTM in Boston, Mass., during the week of June 22, 1958, and of The American Society of Mechanical Engineers in New York City during the week of November 30, 1958. In addition to these meetings the Technical Advisory Committee, consisting of the joint committee officers, executive committee, and panel chairmen, met at Battelle Memorial Inst., Columbus, Ohio, on October 3, 1958, and on March 17, 1959.

Technical Sessions and Papers:

A paper sponsored by the Chemical and Petroleum Panel "Strength of Welded Joints in Low-Alloy Steels at Elevated Temperatures" was presented by W. B. Hoyt at the ASME Petroleum Division meeting in Denver, Colorado, September, 1958.

Two papers sponsored by the joint committee and presented at the ASME Annual Meeting during the week of November 30, 1958 were:

"Comparison of Parameter Methods for Extrapolating High-Temperature Data," by R. M. Goldhoff, General Electric Co.

"Correlation of High-Temperature Creep and Rupture Data," by Hans Conrad, Westinghouse Electric Corp.

At the meeting of the National Association of Corrosion Engineers in Chicago, March, 1959, the joint committee sponsored the presentation of four papers on high-temperature "Metal Dusting" at a Refinery Industry Symposium as follows:

"Corrosion in a Hydrocarbon Conversion System," by F. A. Prange, Phillips Petroleum Co.

"Metal Deterioration in Atmospheres Containing Carbon Monoxide and Hydrogen at Elevated Temperatures," by W. B. Hoyt and R. M. Caughey, M. W. Kellogg Co.

"An Industrial Experience of Severe Metal Wastage Resulting from Burning Methane with Oxygen," by F. Eberle and R. D. Wylie, Babcock & Wilcox Co.

"Preliminary Investigation: The Deteriorating Effect of Carbon Monoxide Atmospheres on the Heat Resisting Alloys," by E. N. Skinner and R. J. Randebaugh, The International Nickel Co.

At the Albany Conference of the ASME Metals Engineering Division, April 30, to May 2, 1959, the joint committee sponsored the following papers:

"Report of Project SP-4 to Steam Power Panel" (High Temperature Tests of SA212, Grade "B" Steel Plate), by J. S. Worth, Bethlehem Steel Co.

"Tubular Stress-Rupture Results," by N. T. Tucker and L. F. Kooistra, Babcock & Wilcox Co.

Membership:

Dr. Richard F. Miller, U. S. Steel Corp., A. L. Penniman, Baltimore Gas and Electric Co., and J. D. Lubahn, General Electric Co., resigned during the year.

New members include J. J. Heger,

---

* Sixty-second Annual Meeting of the American Society for Testing Materials, June 21–26, 1959.

U. S. Steel Corp., Joseph A. Cerow, The Chapman Valve Manufacturing Co., G. E. Klapper, Philadelphia Electric Co., R. M. Van Duzer, Jr., The Detroit Edison Co., R. W. Fenn, Jr., Dow Chemical Co., R. C. Fitzgerald, Baltimore Gas and Electric Co., and Harold Hessing, National Aeronautics and Space Agency.

It is noted with regret that shortly after election to the joint committee Mr. Klapper met an untimely death.

The Technical Advisory Committee has recommended a change in the By-laws of the Committee, making appointment to membership for a three-year period, subject to review for reappointment.

Officers:

At the meeting of December 3, 1958, the following officers were elected for the term expiring June, 1960:

Chairman, J. J. Kanter, Crane Co.

Vice-Chairman, R. A. Baker, Public Service Gas and Electric Co., N. J.

Secretary, H. C. Cross, Battelle Memorial Inst.

Members of Executive Committee: C. L. Clark, Timken Roller Bearing Co., and C. T. Evans, Jr., Universal Cyclops Steel Corp.

Finances:

The status of funds in the committee's account as of January 31, 1959, is given in Table I.

TABLE I.—STATUS OF FUNDS IN THE ACCOUNT OF THE ASTM-ASME JOINT COMMITTEE AS OF JANUARY 31, 1959.

Cash on hand, January 31, 1959				**$17,003.58**
Receipts:				
November, 1958, Westinghouse Electric Corp.			**$6,000.00**	
January, 1959, Edison Electric Inst.			**29,000.00**	**35,000.00**
				$52,003.58
Disbursements, September 30, 1958, to January 31, 1959:				
Postage			**4.85**	
DP-11 and DP-13			**1,139.87**	**1,144.72**
				$50,858.86
Disbursements by projects:				
	Appropriation	***Total Spent Through 1/31/59***	***Balance***	
Data and Publications Panel:				
DP-9, Aluminum and Magnesium Alloys	**$5,200.00**		**$5,200.00**	
DP-10, Physical Properties at Low and High Temperatures	**200.00**	**$122.86**	**77.14**	
DP-11 and DP-13, Continuing Data Survey and Punch Card Tabulation of Data	**3,000.00**	**2,506.90**	**493.10**	
Steam Power Panel:				
SP-5, Use of Type 347 Stainless Steel in Main Steam Piping	**20,000.00**	**...**	**20,000.00**	
SP-6, Use of Austenitic Stainless Steel in Superheater Tube Service	**20,000.00**	**...**	**20,000.00**	
Test Methods Panel:				
Calibration Material	**1,500.00**		**1,500.00**	
Obligated and not spent, January 31, 1959				**47,270.24**
Estimated unallocated balance, January 31, 1959				**$3,588.62**

ACTIVITIES OF PANELS AND PROJECT SUBCOMMITTEES

Finance Committee (N. L. Mochel, chairman):

Action was taken at the December 1958 meeting resulting in the approval of the committee's sponsors for a financial campaign with $150,000 as the goal. This amount includes funds which have already been granted for projects under the Steam Power Panel. A brochure for the purpose of the campaign has been prepared covering the history, objectives, and organization of the committee, and solicitations are under way. The funds anticipated by this campaign are needed to support activities projected by the committee during the next several years.

Panel on Structural Materials for Airframes and Missiles (J. J. Heger, chairman):

This panel was authorized by the Technical Advisory Committee at the October, 1958 meeting and succeeds the Aviation Panel. It held an organizational meeting in December, 1958 and, under its charter, defined its objectives in the following areas:

1. Standardization of testing techniques which may include sponsoring of round-robin testing programs;
2. Correlation of test results with service performance; and
3. The sponsoring of symposia to document information that has been obtained from extensive testing programs conducted by the metal producers and fabricators.

To implement the program, task groups are established as follows:

1. Rapid Heating and Strain Rate Properties,
2. Effect of Temperature on Elastic Constants,
3. Elevated Temperature Compression Properties of Sheet Materials, and
4. Properties of Refractory Metals.

Organization of membership of the panel is proceeding, incorporating that of the former Aviation Panel plus additional personnel necessitated by the altered and expanded scope.

Data and Publication Panel (George V. Smith, chairman):

This panel met on June 24 and again on December 2, 1958, and the status of its activities may be summarized as follows:

Project DP-8, Medium-Carbon, High-Carbon and Plain Carbon Steels, has been concluded with the publication of *ASTM STP No. 228*, "The Report on Elevated Temperature Properties of Chromium Steels," and the project group discharged.

Project DP-9, Aluminum and Magnesium Alloys.—The report on a survey of data evaluated by the personnel of the University of Michigan is at hand for approval of the joint committee for publication.

Project DP-10, Effect of Temperature on Selected Physical Properties of Metals.—A comprehensive survey of physical properties at low and high temperatures on materials has been approved by the joint committee to be undertaken at Battelle Memorial Inst. to the estimated sum of $7500 to be appropriated when funds become available. This survey, which will include steels, stainless steels, super alloys including nickel- and cobalt-base alloys, molybdenum-base alloys, and references to sources of available data on aluminum, titanium, and magnesium-base alloys, anticipates expenditures of the funds over a period of ten months.

Projects DP-11 on Current and Continuing Data Survey and 13 on Punch

Card Tabulation of Data.—A gratis distribution of punch cards incorporating data for 22 materials has been made to some 75 laboratories that had earlier expressed an interest in this cooperative effort. The panel is studying the further enlisting of cooperation of testing laboratories and the problems of making data available.

Gas Turbine Panel (C. T. Evans, Jr., chairman):

This panel held meetings on June 25 and December 1, 1958.

At the June 25 meeting E. C. Bennett of Marquardt Aircraft presented a paper before the panel on a universal high-temperature machine capable of wide variations in strain rates, including simulated missile loads, involving a field of testing which has since come under active study by the Test Methods Panel.

The December 1 meeting was devoted largely to discussion of the future program of the panel, and the matter of proper representation of interests between non-aircraft gas turbines and aviation gas turbines, which falls under the Gas Turbine Panel in realignment of joint committee activities recommended by the Technical Advisory Committee on October 3, 1958.

At the Technical Advisory Committee meeting on March 17, 1959, the resignation of Mr. Evans as panel chairman prompted the authorization of a special study group to appraise gas turbine problems as they relate to the activities of the joint committee.

General Research Panel (J. D. Lubahn, chairman):

This panel held meetings March 11, June 24, and December 1, 1958.

Publication of three surveys has been contracted for by the panel: (1) creep damage (University of Michigan), (2) notch rupture behavior (Syracuse University), and (3) effect of cyclic loading. Publication of these three surveys will be arranged through ASTM in the near future.

At the December meeting a proposal for a comprehensive program of cyclic loading and heating studies was discussed, but no action was taken pending the availability of funds for work of this character.

Mr. Lubahn tendered his resignation as panel chairman at the December meeting.

At the March 17 meeting of the Technical Advisory Committee, action was taken to reconstitute the General Research Panel as an Applied Research Panel, having as its charter such activities as will serve the end of utilizing basic information as it emerges to have its proper effect on engineering practices. It is anticipated that a chairman for the panel will be confirmed at the next joint committee meeting.

Low-Temperature Panel (A. L. Tarr, chairman):

The panel has continued its studies and organization for the accumulation of data on metal and alloys at low temperatures under Project LT-2. The panel is maintaining liaison with related activities progressing under the National Bureau of Standards' Cryogenic Laboratory.

The panel is concerned over its relationship to activities under various other committees working in the fields of brittle fracture and brittle failures. This situation was given considerable review by the Technical Advisory Committee at its recent meeting and it was thought that the panel might make its most effective contributions to the over-all efforts by undertaking projects related to the methodology of defining capacities of materials to resist brittle failure and fracture. Movements are progressing in the field of metal specifications writing toward the transition temperature concept rather than to notch-bar energy absorption values in defining acceptable material, for services where materials with capacity to resist brittle failures are needed. The problems of defining materials on the transition temperature criterion are manifold and are expected to present numerous problems related to the scope of the Low-Temperature Panel.

Chemical and Petroleum Panel (*C. L. Clark, chairman*):

The panel has been credited elsewhere in this report with its participation in the NACE symposium in March, 1959, on high-temperature "metal dusting," and it is anticipated that the papers sponsored by the joint committee will receive early publication after proper review. It is also anticipated that the panel will sponsor further papers and research projects on this somewhat baffling problem of metal dusting which looms so importantly in operations in the petrochemical industries.

The panel is concerned over the stress cracking of heat-exchanger tubing and has under discussion with Subcommittee IX of ASTM Committee A-1 on Steel recommendations for investigations in this area.

The panel is concerned over the problem entailed in the chemical and petroleum industries by the new system of carbon limitations on the 300 series austenitic steels occasioned by ASTM Specifications changes in behalf of the steam power industry through ASME Boiler Code request.

Further work is under way in the area of rupture under high-temperature tests at the weld - base metal interfaces in ferritic alloy steels.

Steam Power Panel (*P. Brister, chairman*):

Project SP-2, Elevated Temperature Properties of Cast Iron.—The final report of this project has been approved as an ASTM Special Technical Publication, *ASTM STP No. 248.*

Project SP-5, The Use of Austenitic Steels in Main Steam Line Service.—This project has been active for some time past and, although several developments have reduced the frequency of cracking in main steam line service, a basic knowledge of the causes of cracking needs yet to be determined. An amplified program to this end has been developed and is being pursued at the University of Michigan under contract by the joint committee. The initial work on this project is carried on under support from International Nickel Co. and Westinghouse Electric Corp. The current greatly expanded scope of the work has been made possible by a grant from the Edison Electric Inst.

Project SP-6, The Use of Austenitic Stainless Steel in Superheater Tube Service.—This project, authorized in June, 1957, to investigate the excessive creep and premature failure experienced with stainless steel superheater tubes, has developed a comprehensive program related to the new provisions adopted in ASTM specifications and the ASME Boiler Code intended to assure adequate high-temperature strength. A contract has been activated at the University of Michigan to carry out a three-year program. The funds for this program, entailing a total expenditure of $60,000, have been granted by Edison Electric Inst.

Project SP-8, Effect of Long-Time Service on Elevated Temperature Properties of Tubes and Pipes.—This project was authorized at the June, 1958 meeting at the request of the Edison Electric Inst. A project committee is charged with defining a scope and aim to be reported at the next panel meeting.

Test Methods Panel (*J. W. Freeman, chairman*):

This panel held meetings June 24 and

December 2, 1958, under Chairman J. J. Kanter. At the March 17, 1959 meeting of the Technical Advisory Committee, Mr. Freeman accepted chairmanship of the panel relinquished by Mr. Kanter upon election as chairman of the joint committee.

Project TM-2, Calibration Specimens for Creep-Rupture Testing.—A heat of type 304 steel (18 per cent chromium, 8 per cent nickel) has been made and judged to have satisfactory characteristics for evaluation and calibration for a creep-rupture specimen bank. Accordingly this material has been placed in the hands of the National Bureau of Standards where testing work is now in progress under contract with the joint committee. It is expected that at an early date the panel will be able to make arrangements for the distribution of calibrated specimens, a service which will probably be handled on a cost-fee basis by ASTM Headquarters.

Through request from the Materials Advisory Board of the National Academy of Sciences, referred by ASTM to the Test Methods Panel, a project has been undertaken for the standardization of testing procedures needed for airframe and missile materials. A task force was formed under the panel and, with cooperation from the Aircraft Industries Assn. through its Aircraft Research and Testing Committees, has established the necessary additional representation to proceed effectively with the undertaking. This task force met in February at Ann Arbor, Mich., and prepared a draft of a proposed ASTM recommended practice for rapid heating-rate and rapid strain-rate tension tests of metallic materials at elevated temperatures, to be submitted for action at the June, 1959 meeting of the joint committee for recommendation to ASTM as a tentative recommended practice.

Committee on International Conference on Elevated Temperature Behavior of Materials.—At the time of the December ASME meeting, the joint committee was approached by the Metals Engineering Division with a proposal to sponsor jointly an International Conference. Two planning meetings have been held so far between representatives of the two sponsor groups in preparation for the conference.

This report has been submitted to letter ballot of the committee, which consists of 32 members; 18 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. J. KANTER,
Chairman.

HOWARD C. CROSS,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, the Joint Committee on Effect of Temperature on the Properties of Metals presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Recommended Practice for:

Conducting Creep and Creep Rupture Tension Tests of Metallic Materials Under Conditions of Rapid Heating and Short Times (E 150 – 59 T), and

Tension Tests of Metallic Materials at Elevated Temperatures with Rapid Heating and Conventional or Rapid Strain Rates (E 151 – 59 T).

These recommendations were accepted by the Standards Committee on September 15, 1959, and the new tentative recommended practices appear in the 1959 Supplement to the Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-1

ON

CEMENT*

Committee C-1 on Cement held two meetings during the year: in Boston, Mass., on June 27, 1958, and in Lafayette, Ind., on December 2, 1958.

The committee records with sorrow the deaths of Robert F. Blanks, a member since 1940, who at the time of his death represented the Great Western Aggregates, Inc., and Clarence Lamoreaux, a member since 1949, who at the time of his death represented the Buffalo Slag Co., Inc.

At the 1958 Annual Meeting of the Society, M. A. Swayze received the ASTM Award of Merit.

In recognition of his long association with Committee C-1 and his contributions to its work, D. G. Miller was made an Honorary Member of the committee.

NEW TENTATIVES

The committee recommends for publication as tentative the Method of Test for Fineness of Hydraulic Cement by the No. 325 Sieve as appended hereto.[1]

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Method for Mechanical Mixing of Hydraulic Cement Mortars of Plastic Consistency (**C 305 - 58 T**)[2] and continuation of the method as tentative.

Title.—Revise the title to read "Tentative Methods for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency."

Section 1.—Change to read as follows: "1. This method covers the mechanical mixing of hydraulic cement pastes and mortars of plastic consistency."

Section 4.—Change to read as follows:

> 4. The materials, their proportions and quantities shall conform to the requirements contained in the particular method of test for which the paste or mortar is being prepared.

New Section.—Add a new Section 5 entitled "Procedure for Mixing Pastes," to read as follows:

> 5. Place the dry paddle and the dry bowl in the mixing position in the mixer. Then introduce the materials for a batch into the bowl and mix in the following manner:
>
> (*1*) Place all the mixing water in the bowl.
> (*2*) Add the cement to the water and allow 30 sec for the absorption of the water.
> (*3*) Start the mixer and mix at slow speed (140 ± 5 rpm) for 30 sec.
> (*4*) Stop the mixer, and quickly scrape down into the batch within 15 sec any paste that may have collected on the bowl.
> (*5*) Start the mixer at medium speed (285 ± 10 rpm) and mix for 1 min.

Section 5.—Change the title to read "Procedure for Mixing Mortars" and renumber as Section 6.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

---

* **Sixty-second Annual Meeting of the Society, June 21-26, 1959.**

[1] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 4.

[2] **1958 Book of ASTM Standards, Part 4.**

Standard Specifications for Portland Cement (C 150 – 56),[2] and Standard Specifications for Air-Entraining Portland Cement (C 175 – 56):[2]

Table I.—Add the following Note below Table I in each specification:

NOTE.—Cement containing not more than 0.60 per cent alkalies, calculated as per cent of Na_2O + 0.658 times per cent of K_2O, may be specified when the cement is to be used in concrete with aggregates that may be deleteriously reactive. Reference is made to Specifications for Concrete Aggregates (ASTM Designation: C 33) for suitable criteria of deleterious reactivity.

Standard Method of Test for Fineness of Portland Cement by Air Permeability Apparatus (C 204 – 55):[2]

Section 2(d).—Change the first sentence to read "The plunger shall consist of a phenolic laminate and shall fit into the cell with a clearance of not more than 0.1 mm."

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Air Content of Hydraulic Cement Mortar (**C 185 – 58 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Masonry Cement (C 91 – 58):[2]

In order to provide a more comprehensive explanation and an example for proportions for mortar, the following revisions are recommended:

Section 20(a).—Delete the present Paragraph (*a*) and replace with the following:

20. (*a*) *Proportions for Mortar.*—Mortar for the compressive strength test (Note 1) shall be proportioned to contain the weight of cement, in grams, equal to six times the printed bag weight in pounds and 1440 g of sand. The sand shall consist of 720 g of graded Ottawa sand and 720 g of 20–30 Ottawa sand (Note 2). The quantity of water, measured in milliliters, shall be such as to produce a flow of 110 ± 5 as determined by the flow table.

NOTE 1.—Mortars for the water retention and air entrainment tests are prepared in exactly the same way as the mortar for the compressive strength test.

NOTE 2.—If the printed bag weight, in pounds, is 70, the mortar shall contain 420 g of cement. The specified mortar proportions are approximately the 1:3 nominal proportions, by volume, commonly specified for construction, as indicated in the following calculation: If a bag of masonry cement contained 1 cu ft of cement, and 1 cu ft of loose damp sand contained 80 lb of dry sand,

$$C = \frac{1440}{240} \times \text{weight per bag (lb)}$$

$$= 6 \times \text{weight per bag (lb)}$$

where C is the number of grams of cement to be used in the mortar with 1440 g of sand, and 240 is the weight in pounds of dry sand in 3 cu ft of loose damp sand.

Standard Specifications for Portland Cement (C 150 – 56):[2]

In order to permit the use of the optimum amount of SO_3 in the manufacture of Type III cement, the following revision is recommended:

Table 1.—Opposite the words "When $3CaO \cdot Al_2O_3$ is more than 8 per cent, max, per cent" and in the column for Type III, change "3.0" to "4.0."

Standard Methods of Test for Autoclave Expansion of Portland Cement (C 151 – 58):[2]

In order to effect some helpful changes in requirements for apparatus and to provide new information concerning the number of test specimens, the following revisions are recommended:

Section 2.—In Paragraph (*c*) delete the first sentence and replace with the following two sentences: "Molds may be either single or double (Figs. 1 and 2). Molds shall provide for 1 by 1-in. test specimens of 10-in. effective gage length (Note)."

In Paragraph (*e*), in the second sentence, change "1 to $1\frac{1}{4}$ hr" to read "45 to 75 min."

Section 4.—Add a new Paragraph 4 (*a*) to read as follows; relettering subsequent paragraphs accordingly:

> 4. (*a*) *Number of Test Specimens.*—One test specimen shall be made except in the event of a retest when the number of test specimens shall be three as provided for in Section 7.

Standard Specifications for Air-Entraining Portland Cement (C 175 – 56):[2]

In order to permit the use of the optimum amount of SO_3 in the manufacture of Type IIIA cement, the following revision is recommended:

Table I.—Opposite the words "When $3CaO \cdot Al_2O_3$ is more than 8 per cent, max, per cent" and in the column under Type IIIA, change "3.0" to "4.0."

Standard Method of Test for Tensile Strength of Hydraulic Cement Mortars (C 190 – 58):[2]

In order to promote uniformity in the reporting of test results and to incorporate a needed requirement on mold thickness, the following revisions are recommended:

Section 2(e).—Change the last sentence of this paragraph to read as follows:

> The dimensions of the briquet molds shall conform to the following requirements: width of mold, between inside faces, at waist line of briquet, 1 in. with permissible variations of plus or minus 0.01 in. for old molds and plus or minus 0.005 in. for new molds; thickness of molds measured at point of greatest thickness on either side of mold at waist line, 1 in. with permissible variations of plus 0.004 in. and minus 0.002 in. for new molds, and minus 0.02 in. for molds in use.

New Section.—Add a new Section 11 to read as follows:

> 11. The tensile strength of all acceptable test specimens (see Section 10) made from the same sample and tested at the same period shall be averaged and reported to the nearest 5 psi.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Flow Table for Use in Tests of Hydraulic Cement (**C 230 – 57 T**).

Tentative Methods of Test for:

Flexural Strength of Hydraulic Cement Mortars (**C 348 – 57 T**),

Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) (**C 349 – 57 T**), and

False Set of Portland Cement (**C 359 – 56 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standard and tentatives:

Standard Methods of Chemical Analysis of Portland Cement (C 114 – 58):[2]

Section 20(a).—In the second sentence of the Note, change "section 19" to read "section 30."

Section 34.—In the italicized heading, change "*(0.18N)*" to read (*"approximately 0.18N).*"

Tentative Methods of Sampling Hydraulic Cement (C 183 – 58 T):[2]

Section 3(c).—In the heading and in the fourth sentence, change "tube sampler" to read "slotted tube sampler."

Tentative Specifications for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 – 58 T):[2]

Section 1.—Delete the Note at the end of this section.

Tentative Method of Test for False Set of Portland Cement (C 359 - 56 T):[3]

Section 2(c).—Change to read "*Containers*, approximately 2 by 2 by 6 in. inside dimension (Fig. 1)."

Section 4(d).—In the third sentence insert the word "wet" before the word "mixing."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Working Committee on SO_3 Content.—A special report, entitled "Short-Time Tests of Mortars for Controlling SO_3 in Portland Cement at Optimum Value," is appended hereto.[4]

This report has been submitted to letter ballot of the committee, which consists of 86 voting members; 65 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

W. J. MCCOY,
Secretary.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[4] See p. 349.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards the following recommendation:

Revision of Tentative Method of Test for:

Compressive Strength of Hydraulic Cement Mortar (Using Portions of Prisms Broken in Flexure) (C 349 - 57 T).

This recommendation was accepted by the Standards Committee on October 27, 1959, and the revised tentative method appears in the 1959 Supplement to Book of ASTM Standards, Part 4.

APPENDIX I

SHORT-TIME TESTS OF MORTARS FOR CONTROLLING SO_3 IN PORTLAND CEMENT AT OPTIMUM VALUE

REPORT OF WORKING COMMITTEE ON SO_3 CONTENT OF ASTM COMMITTEE C-1 ON CEMENT

A cooperative test program was conducted by the Working Committee on SO_3 Content during 1957 with the objective of determining whether short-time tests for compressive strength and expansion in water and contraction in air of Ottawa sand mortars could be used satisfactorily as a means of determining the relationship of the SO_3 content of cement to its optimum value.

The laboratories participating in the test program were:

California Division of Highways, Sacramento, Calif.,
Ideal Cement Co., Fort Collins, Colo.,
Medusa Portland Cement Co., Wampum, Pa.,
Portland Cement Assn., Chicago, Ill., and
Universal Atlas Cement Co., Buffington, Ind.

The order of listing these participants is not the same as the order in the presentation of data.

Four portland cements were prepared and shipped to the laboratories: two of type III, one of type I, and one of type II.[1] The cements were in the following classifications are to chemical composition:

Cement No. 1.—Type III, high C_3A, low alkali;
Cement No. 2.—Type III, low C_3A, low alkali;
Cement No. 3.—Type I, high C_3A, high alkali; and
Cement No. 4.—Type II, low C_3A, high alkali.

The chemical analysis and specific surface of these cements are given in Table I. The cements were selected on the basis of preliminary tests to have SO_3 contents below optimum. Tests made in this study indicate that the cements were deficient in SO_3 by 0.40 to 2.15 per cent relative to optimum.

BASIS OF TEST PROGRAM

It has been found that in mortars containing portland cements that are relatively low in SO_3, progressive additions of gypsum to the cement produce decreasing contraction in air until a minimum contraction is reached. Further additions of gypsum produce increasing contraction. Similarly, additions of gypsum increase the strength of mortars up to a maximum value and further additions produce decreasing strength. The percentage of SO_3 that results in the least contraction or highest strength in a given cement is termed the optimum SO_3 content.

Curves representing the relationship

---

[1] Specifications for Portland Cement (C 150), 1958 Book of ASTM Standards, Part 4.

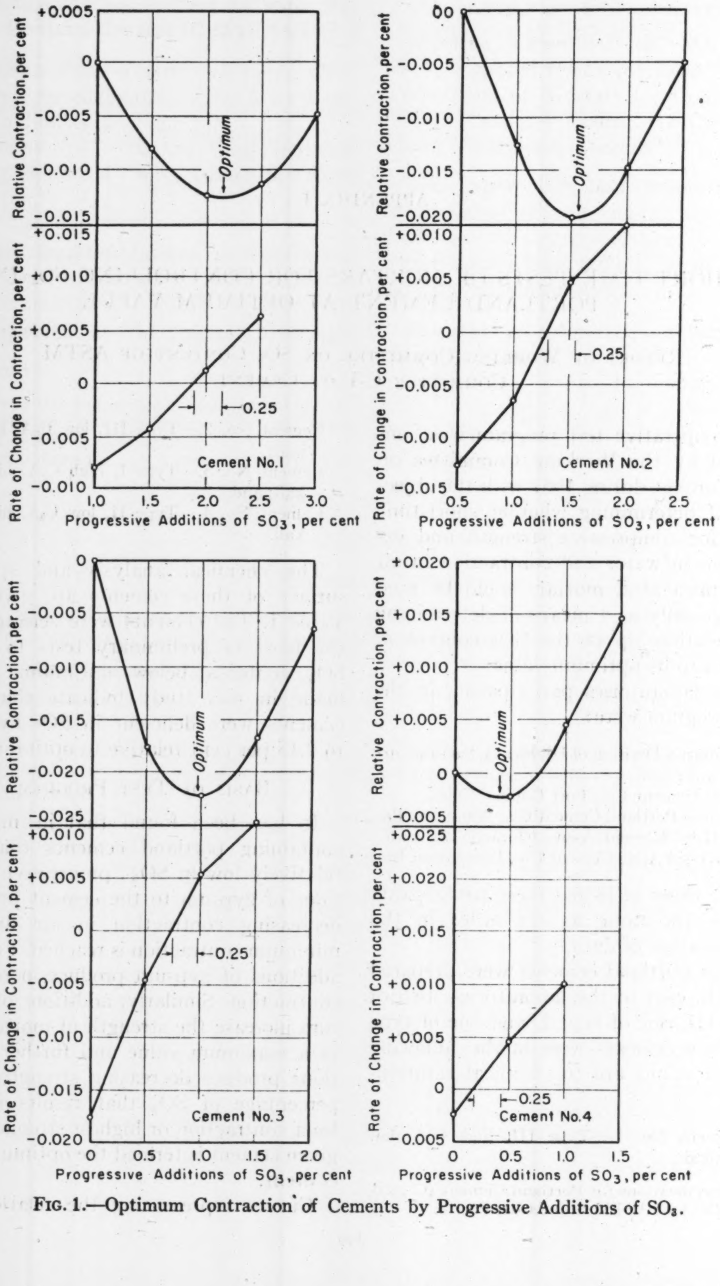


FIG. 1.—Optimum Contraction of Cements by Progressive Additions of SO_3.

between SO_3 content and contraction or strength vary somewhat in shape between cements, but in the region of optimum SO_3 the variation in shape is not great. Figures 1 to 4 illustrate the relationships found with the cements under study. The change in magnitude of contraction or strength produced by a suitable known addition of gypsum provides information on the relationship of the SO_3 in the original cement to its optimum value.

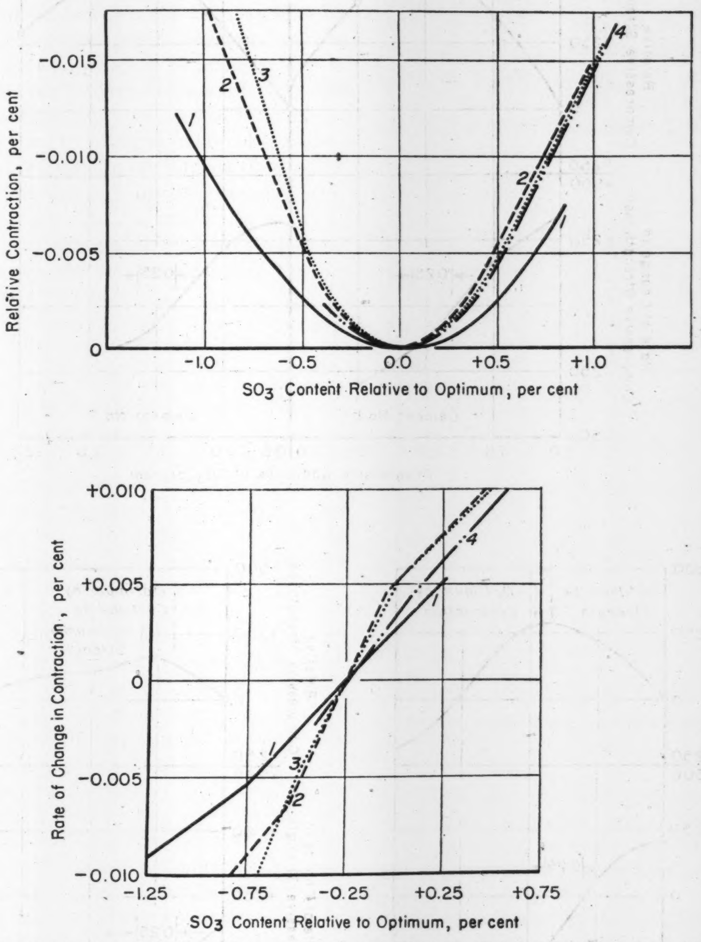


FIG. 2.—Curves of the Four Cements Plotted so That the Point of Optimum Coincides.

When the SO_3 content is increased to about 0.75 per cent above its optimum value, significant increases in the expansion in water result.

The plan of the test program was based on the relationships shown in Figs. 1 to

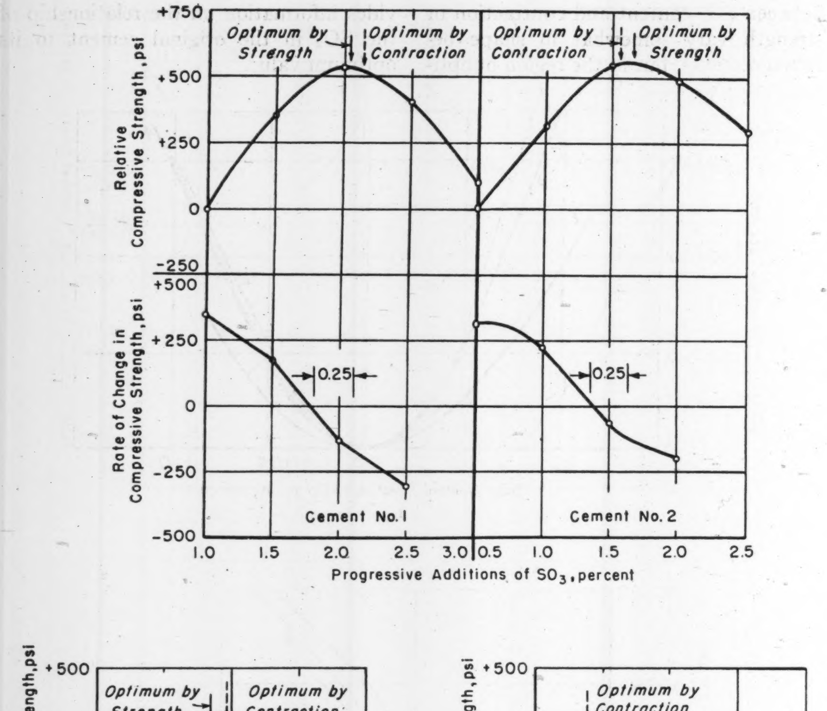


FIG. 3.—Compressive Strength *Versus* SO_3 Content.

4. The methods require the testing of specimens made from mortars containing the original cement and the same cement blended with pulverized gypsum in an amount to increase the SO_3 content by 0.5 percentage points. Results of the tests are known after 24 hr for strength, 3 days for expansion, and 7 days for contraction.

The utility of these tests as a manufacturing guide or as information to the consumer depends on the precision with which they can be made. A study of precision, repeatability, and reproducibility was the main objective of the cooperative program.

GENERAL SCOPE OF TESTS

Preliminary information indicated that measurable changes in the physical properties of mortars could be obtained by varying the SO_3 content by progressive additions of 0.5 per cent. Therefore, pulverized gypsum was added and blended with the cements to provide increments at this interval. Gypsum having the properties shown in Table I was used by all laboratories.

In order to evaluate the results of the tests, it was necessary to establish good values of optimum SO_3 content of each cement. For this reason the cements were blended with gypsum to provide five progressive additions of SO_3 in the range of 1 per cent below to 1 per cent above optimum. Cement No. 4, however, was received with an SO_3 content of about 0.5 per cent below optimum. The range for this cement extended from this value to 1.5 per cent above optimum.

DETAILED SCOPE OF TESTS

The complete schedule of tests included gathering data on the expansion and contraction, compressive strength, and calcium sulfate in hydrated cement mortar.

Expansion and Contraction:

Expansion was determined by storing specimens in water for 48 hr followed by contraction in air for 4 days. Each test series consisted of four 1- by 1-in. by 10-in. gage length bars molded from a batch of 1 part cement to 2 parts (by weight) graded Ottawa sand mixed as specified in ASTM Method C 109, Section 8.[2] Bars were removed from the molds after 24 hr moist storage, at which time the initial measurement of length was made. The bars were then stored in water at 74.3 ± 3 F for 48 hr, after which they were again measured for length and the percentage of expansion was computed. The length at the conclusion of the expansion test was taken as the initial length for the contraction test. The bars were stored in a specially

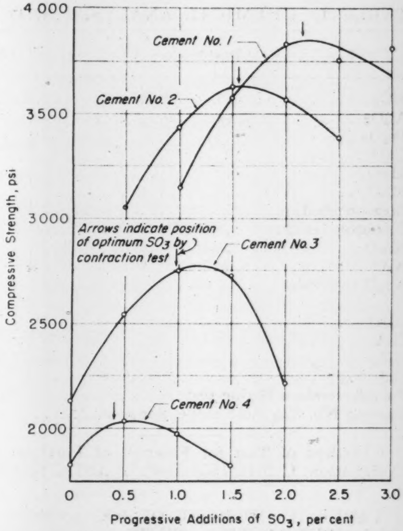


FIG. 4.—Compressive Strength Data of Laboratory No. 5.

---

[2] 1958 Book of ASTM Standards, Part 4.

designed cabinet described in the Method of Test for Expansion in Water and Contraction in Air of Portland-Cement Mortar appended to this report.[3] The cabinet was installed in a room maintained at a temperature between 68 and 81.5 F. Relative humidity within the cabinet was of the order of 50 per cent at equilibrium.

Table II indicates the number of test series made with each cement. Tables III and IV list the average expansion and contraction of each cement.

Except for cement No. 4, this schedule provided progressive additions in SO_3 content from about minus 1 to plus 1 per cent relative to optimum.

TABLE I.—CHEMICAL ANALYSES AND FINENESS OF CEMENTS AND GYPSUM.

Cement	No. 1	No. 2	No. 3	No. 4	Gypsum
SiO_2	21.0	21.8	21.0	22.5	0.20
Al_2O_3	6.3	4.4	5.9	4.5	0.20
Fe_2O_3	2.2	3.8	2.8	4.5	
CaO	65.4	64.3	63.1	64.4	33.0
MgO	1.3	2.2	2.7	1.0	0.2
SO_3	1.71	1.49	2.28	1.70	46.4
Loss on ignition	1.3	1.10	0.85	0.62	...
Insoluble residue	0.22	0.19	0.18	...	...
Na_2O	0.10	0.35	0.53	0.66	...
K_2O	0.64	0.17	0.69	0.23	...
Na_2O equivalent	0.52	0.46	0.98	0.81	...
C_4AF	7	12	8	14	...
C_3A	13	5	11	4	...
C_3S	56	57	46	49	...
C_2S	18	20	25	28	...
Specific surface Blaine test[a]	4690	5560	3290	2740	...
Passing No. 325 mesh sieve, per cent	...	...	...	...	93

[a] Method of Test for Fineness of Portland Cement by Air Permeability Apparatus (ASTM Designation: C 204), 1958 Book of ASTM Standards, Part 4.

TABLE II.—NUMBER OF EXPANSION AND CONTRACTION TESTS FOR EACH CEMENT.

Progressive additions SO_3, per cent	0	0.5	1.0	1.5	2.0	2.5	3.0
Cement No. 1	...	...	5	5	3	3	3
Cement No. 2	...	5	5	3	3	3	...
Cement No. 3	5	5	3	3	3	...	...
Cement No. 4	5	5	3	3	3	...	...

Compressive Strength:

Compressive strength was determined by Method C 109[2] using a six-cube batch. Tests were made at 1 and 3 days on type III cements and at 1 and 7 days on type I and type II cements. Compressive strength results at 3 and 7 days are not presented in this report because they

TABLE III.—AVERAGE EXPANSION OF EACH CEMENT.

Values shown are the arithmetical means of all acceptable results, in per cent.

Progressive additions of SO_3, per cent	0	0.5	1.0	1.5	2.0	2.5	3.0
Cement No. 1	...	...	0.0069	0.0072	0.0067	0.0076	0.0157
Cement No. 2	...	0.0058	0.0054	0.0057	0.0074	0.0122	...
Cement No. 3	0.0059	0.0045	0.0039	0.0061	0.0197	...	...
Cement No. 4	0.0050	0.0057	0.0085	0.0114	0.0140	...	...

---

[3] See p. 366.

were found to be less indicative of optimum SO_3 content than were the 24-hr strengths. Three test series were made at each of the increments of SO_3 indicated in Table II. Table V shows the average compressive strength for each cement at the various progressive additions of SO_3.

Calcium Sulfate in Hydrated Cement Mortar:

These tests were made in accordance with ASTM Method C 265 – 57 T,[2] except that tests were made at 18 hr as well as the specified 24 hr.

TABLE IV.—AVERAGE CONTRACTION OF EACH CEMENT.

Values shown are the arithmetical means of all acceptable results, in per cent.

Progressive additions of SO_3, per cent	0	0.5	1.0	1.5	2.0	2.5	3.0
Cement No. 1	...	...	0.0695	0.0604	0.0582	0.0572	0.0646
Cement No. 2	...	0.0721	0.0568	0.0521	0.0564	0.0665	...
Cement No. 3	0.0644	0.0478	0.0437	0.0484	0.0589	...	...
Cement No. 4	0.0337	0.0305	0.0376	0.0483	0.0528	...	...

TABLE V.—AVERAGE COMPRESSIVE STRENGTH OF EACH CEMENT.

Values shown are the arithmetical means of all results reported by Laboratories Nos. 1 to 4 inclusive at the age of 24 hr, in psi.

Progressive additions of SO_3, per cent	0	0.5	1.0	1.5	2.0	2.5	3.0
Cement No. 1	...	...	1922	2224	2400	2273	2026
Cement No. 2	...	1560	1875	2105	2038	1839	...
Cement No. 3	1237	1451	1509	1268	1052	...	...
Cement No. 4	955	1137	1099	1001	917	...	...

Single test series were made with additions of SO_3 as indicated in Table VI.

Execution of Tests:

Laboratories Nos. 2 and 3 completed the entire schedule of tests. Laboratory No. 1 also completed the schedule but used a controlled room in place of the special cabinet for the contraction tests. Laboratory No. 4 completed two test rounds of the expansion-contraction and compressive strength tests. Laboratory No. 5 varied the designated procedure in a number of ways. The results from this laboratory were used in establishing the best values of optimum SO_3 content, but they could not be pooled with the others in computations of the precision of the tests.

The SO_3 content of each cement was reported by the supplier. Blends containing additional quantities of SO_3 were calculated by the following formula:

$$X = \frac{A - B}{C - B} \times 100,$$

$$Y = 100 - X$$

where:

X = per cent gypsum in the blend,
Y = per cent cement in the blend,
A = per cent desired SO_3 content of the blend,
B = per cent SO_3 content of the cement, and
C = per cent SO_3 content of the gypsum.

All laboratories used the same designated percentage of water in expansion-contraction and compressive strength tests of mortars containing cements Nos. 1, 2, and 3. Each laboratory independently predetermined the water required in mortars containing cement No. 4. In all cases the water was held constant for all increments of SO_3. Flow tests indicated that the addition of gypsum produced only a minor change in consistency. A constant amount of water was used in the test for calcium sulfate in hydrated cement mortar as prescribed in the standard method.

The cement-gypsum blends were prepared in individual lots as required for each batch of mortar. The required amount of gypsum was weighed to the nearest 0.1 g. Cement was then added to bring the total weight to the required batch quantity. The weighed mixture was then transferred to a bowl and mixed with a rubber spatula preparatory to adding it to the water in the mechanical mixer.

TABLE VI.—PROGRESSIVE ADDITIONS OF SO_3 FOR SINGLE SERIES OF TESTS.

Additions of SO_3, per cent	0	0.5	1.0	1.5	2.0	2.5	3.0
Cement No. 1	...	...	X	...	X	...	X
Cement No. 2	...	X	...	X	...	X	...
Cement No. 3	X	...	X	...	X	...	...
Cement No. 4	X	...	X	...	X	...	...

OPTIMUM SO_3 CONTENT OF EACH CEMENT

The best value of optimum SO_3 for contraction was determined by pooling the data reported by the five laboratories. Differences in contraction produced by each progressive addition of SO_3 in each test round were calculated. The results were then analyzed statistically by the control chart method. The average results of tests that were in control are tabulated in Table VII. Except in one instance, at least four of the five laboratories were found to be in control for each increment of each cement. The cumulative change in contraction was calculated and used to plot the curves shown in Fig. 1. Figure 1 also shows the rate of change in contraction for each increment of SO_3. The SO_3 content at which these curves intersect the ordinate of zero change is very nearly 0.25 per cent below the optimum value. A slight discrepancy results when the upper plotted curves are not symmetrical about the axis of optimum SO_3 but the error is not greater than 0.05 per cent SO_3. The arrows in the upper curves of Fig. 1 indicate the position of optimum SO_3.

The curves of the four cements are plotted in Fig. 2 so that the point of optimum coincides. The rate of change curves in the lower part of Fig. 2 are plotted so that the points of zero change in contraction coincide.

The changes in compressive strength produced by increases of 0.5 per cent SO_3 as reported by four laboratories were calculated in the same manner as were the changes in contraction. The results are given in Table VIII. Curves of compressive strength *versus* SO_3 content are shown in Fig. 3, using the standard mortar of Section 8, Method C 109. The precision of the compressive strength test is not as high as that of the contraction test. Consequently, the curves of Fig. 3 could not be plotted with the same degree of confidence. The optimum SO_3 content for strength was determined in the same manner as that for contraction. The arrows in the upper curves of Fig. 3 indicate the optimum determined by both methods. It will be noted that there is no serious discrepancy. This finding is of importance since a number of investigators have reported different values of optimum for contraction and strength. Their conclusions, however, were not based on data as comprehensive as those obtained in this cooperative study.

The compressive strength results determined by laboratory No. 5 could not be pooled with the others because they were made on 1:2 mortar instead of the designated 1:2.75 mortar. The results of laboratory No. 5 are plotted in Fig. 4.

TABLE VII.—SUMMARY OF DATA USED IN ESTABLISHING OPTIMUM SO₃ FOR MINIMUM CONTRACTION.

Values shown are change in contraction in per cent produced by increasing the SO_3 content 0.5 per cent above the percentage shown in each column.

Decreasing contraction indicated as minus, increasing contraction indicated as plus.

Progressive additions of SO_3, per cent	0	0.5	1.0	1.5	2.0	2.5
Cement No. 1						
Number of laboratories in control	...	...	4	3	4	5
Average change in contraction			−0.0080	−0.0044	+0.0011	+0.0065
Cumulative change			−0.0080	−0.0124	−0.0113	−0.0048
Standard deviation			0.0022	0.0010	0.0021	0.0023
Cement No. 2						
Number of laboratories in control	...	5	4	4	5	...
Average change in contraction		−0.0127	−0.0066	+0.0046	+0.0098	
Cumulative change		−0.0127	−0.0193	−0.0147	−0.0049	
Standard deviation		0.0053	0.0017	0.0023	0.0028	
Cement No. 3						
Number of laboratories in control	5	5	4	5	...	...
Average change in contraction	−0.0176	−0.0046	+0.0054	+0.0103		
Cumulative change	−0.0176	−0.0222	−0.0168	−0.0065		
Standard deviation	0.0031	0.0025	0.0013	0.0035		
Cement No. 4						
Number of laboratories in control	4	5	4	...	...	...
Average change in contraction	−0.0022	+0.0068	+0.0101			
Cumulative change	−0.0022	+0.0046	+0.0147			
Standard deviation	0.0013	0.0019	0.0012			

NOTE.—Laboratories in control were determined by control chart method of analysis. Individual laboratories reported from two to five test series. Computations were made to compensate for differences in number of test series. Standard deviations were computed only from results that were in control.

TABLE VIII.—SUMMARY OF DATA USED IN ESTABLISHING OPTIMUM SO₃ FOR MAXIMUM COMPRESSIVE STRENGTH.

Values shown are change in compressive strength in psi produced by increasing the SO_3 content 0.5 per cent above the percentage in each column for 24 hr.

Decreasing strength indicated as minus, increasing strength indicated as plus.

Progressive additions of SO_3, per cent	0	0.5	1.0	1.5	2.0	2.5
Cement No. 1						
Number of laboratories in control	...	...	3	4	4	3
Average change in strength			+349	+176	−136	−301
Cumulative change			+349	+525	+389	+88
Standard deviation			70	56	128	52
Cement No. 2						
Number of laboratories in control	...	4	4	4	4	...
Average change in strength		+316	+229	−66	−198	
Cumulative change		+316	+545	+479	+281	
Standard deviation		72	64	54	72	
Cement No. 3						
Number of laboratories in control	4	4	4	4	...	...
Average change in strength	+215	+62	−250	−216		
Cumulative change	+215	+277	+27	−189		
Standard deviation	60	61	63	89		
Cement No. 4						
Number of laboratories in control	2	4	3	4	...	...
Average change in strength	+152	−46	−35	−84		
Cumulative change	+152	+106	+71	−13		
Standard deviation	35	39	55	51		

The arrows of these curves represent the optimum SO_3 content for contraction as derived from the test data of the five laboratories. It will be noted that this optimum value is not out of line with the indications of the strength data obtained by laboratory No. 5 using the richer mortar.

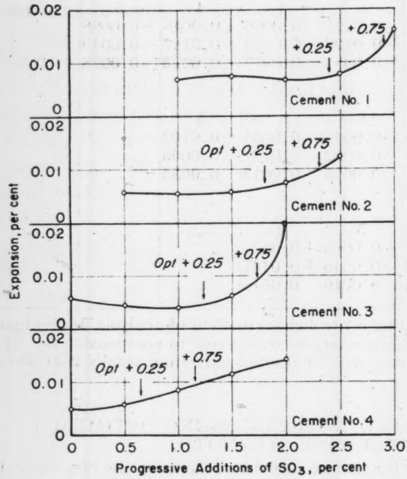


FIG. 5.—Effect of SO_3 Content on Expansion of the Four Cements.

The effect of SO_3 content on expansion is shown in Fig. 5. The data were obtained by pooling the results of the five laboratories. The arrows in these charts show SO_3 contents of 0.25 and 0.75 percentage points greater than the optimum percentage as determined by the contraction tests. It will be noted that a sharp increase in expansion results when the SO_3 content exceeds about +0.75 per cent relative to optimum. The expansion test, therefore, may provide an important safeguard against a gross excess of SO_3 in the cement.

For reasons that will become apparent later in this discussion, *absolute* values of expansion are of greater significance than are the *changes* produced by the progressive additions of SO_3. This report does not contain a table showing the differences in expansion produced by these additions of SO_3.

PRECISION OF THE TESTS

The two measures of precision that are considered are those of repeatability and reproducibility. These measures have been computed in accordance with the Proposed Recommended Practices for Applying Precision Data Given in ASTM Methods for Petroleum Products and Lubricants.[5] These definitions are:

Repeatability is a quantitative measure of the variability associated with a single operator in a given laboratory, generally with the same apparatus and with a small interval of time. It is defined as the greatest difference between two single and independent results that can be considered acceptable (not significantly different) at the 95 per cent probability level (for methods referring to this recommended practice).

Reproducibility is a quantitative measure of the variability associated with operators working in two different laboratories. It is defined as the greatest difference between a single result obtained in one laboratory and a single result obtained in another laboratory that need not be considered suspect (significantly different) at the 95 per cent probability level (for methods referring to this recommended practice).

In these cooperative tests the reported values of expansion and contraction are the average of four individual specimens made from a batch of mortar. When the average result of a group of specimens is treated as a single observation, it is customary to adopt some criterion for eliminating suspicious results. In these cooperative tests the standard deviation of the individual specimens was computed for each test round. A study has been made of these standard deviations for the purpose of determining the limits of variability that can reasonably be expected.

---

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 379 (1953).

An appropriate value of standard deviation for the expansion test is considered to be 0.0025. The number of tests made by each laboratory and the percentage of results that did not exceed this value of standard deviation is shown below:

Laboratory	Number of Tests	Per Cent Meeting Criterion
No. 1.........	90	100
No. 2.........	88	95
No. 3.........	86	56
No. 4.........	44	98

An appropriate value of standard deviation for the contraction test is considered to be 0.0030. The number of tests made by each laboratory and the percentage of results that did not exceed this value of standard deviation is shown below:

Laboratory	Number of Tests	Per Cent Meeting Criterion
No. 1.........	90	94
No. 2.........	88	97
No. 3.........	86	67
No. 4.........	44	98

In the computation of indices of precision, only those results which met the above criteria have been included.

TABLE IX.—PRECISION OF COMPRESSIVE STRENGTH TESTS.

Values are in units of psi computed from change in strength after 24 hr set, produced by increasing the SO_3 content 0.5 per cent above the additions shown in each column.

Progressive additions of SO_3, per cent	0	0.5	1.0	1.5	2.0	2.5
Cement No. 1						
Repeatability....................	...	...	209	200	360	257
Reproducibility..................	...	...	300	111	406	159
Cement No. 2						
Repeatability....................	...	215	175	167	165	...
Reproducibility..................	...	204	177	149	259	...
Cement No. 3						
Repeatability....................	213	209	221	282	...	...
Reproducibility..................	129	131	137	248	...	...
Cement No. 4						
Repeatability....................	121	116	132	144	...	...
Reproducibility..................	570	162	381	98	...	...

Average repeatability.. 199 psi
Average reproducibility.. 226 psi

Average repeatability when SO_3 content is at approximate optimum.................. 216 psi
Average reproducibility when SO_3 content is at approximate optimum................ 214 psi

Similar criteria were not applied to the tests for compressive strength because they were made in accordance with Method C 109[2] and this method provides rules for rejecting suspected results.

Precision of Compressive Strength Test:

Considering first the compressive strength data at 24 hr, the addition of gypsum to increase the SO_3 content by 0.5 per cent when the cement contained optimum SO_3 produced reductions in strength as follows:

Cement No. 1	200 psi
Cement No. 2	180 psi
Cement No. 3	250 psi
Cement No. 4	40 psi

These values have been scaled from carefully plotted curves similar to those shown in Fig. 3. Values of repeatability and reproducibility as shown in Table IX are 216 and 214 psi, respectively, for the 24-hr compressive strength tests. These measures are nearly as large as the value measured for cement No. 3 and are over five times as large as the measured value for cement No. 4. It does not appear, therefore, that compressive strength results can be used advantageously to indicate the relationship of SO_3 content to optimum unless many more than two batches of mortar are tested.

Precision of Contraction Test:

Computed values of repeatability and reproducibility for expansion and contraction are given in Tables X and XI. There is considerable variation in values for different cements and different additions of SO_3. These variations are believed to result from the relatively small number of results that were available, in comparison with the ideal of an infinite number. For this reason the average values of all cements is considered to be more nearly correct. It is evident that the precision is better when the SO_3 content is near the optimum.

In this study, values of reproducibility are of greater interest than those of repeatability. The average reproducibility of the difference in contraction between cement at optimum SO_3 and at optimum plus 0.5 per cent SO_3 is 0.0046

TABLE X.—PRECISION OF EXPANSION TESTS.

Values are for direct recorded measurements, not for differences between progressive additions of SO_3.

Progressive additions of SO_3, per cent	1.0	1.5	2.0	2.5	3.0
Cement No. 1					
Repeatability	...	...	0.0036	0.0022	0.0178
Reproducibility	...	...	0.0033	0.0067	0.0125
Cement No. 2					
Repeatability	...	0.0085	0.0104	0.0106	...
Reproducibility	...	0.0066	0.0020	0.0049	...
Cement No. 3					
Repeatability	0.0040	0.0049	0.0068	...	...
Reproducibility	0.0038	0.0069	0.0149	...	...
Cement No. 4					
Repeatability	0.0024	0.0051	0.0067	...	...
Reproducibility	0.0027	0.0085	0.0223	...	...

Average reproducibility for absolute values of expansion are:

At optimum SO_3	0.0041
At optimum SO_3 + 0.25 per cent SO_3	0.0050 (interpolated)
At optimum SO_3 + 0.50 per cent SO_3	0.0060
At optimum SO_3 + 0.75 per cent SO_3	0.0076 (interpolated)
At optimum SO_3 + 1.00 per cent SO_3	0.0102

These indices, while large with respect to the values to be measured, are more favorable than those of the compressive strength indices.

It should be noted that they represent

the range or greatest difference between single acceptable test results. The departure of a single result from the true value should not be more than one half of the computed value of reproducibility.

Because of the statistical methods used in deriving the values shown in the curves of Figs. 1, 3, and 5, it can be assumed with considerable confidence that these values are very close to the true values and that single results should not depart from them by more than one half the value of reproducibility.

TABLE XI.—PRECISION OF CONTRACTION TESTS.

Values are in units of per cent contraction computed from change in contraction produced by increasing the SO_3 content 0.5 per cent above the progressive additions shown in each column.

Progressive additions of SO_3, per cent	0	0.5	1.0	1.5	2.0	2.5
Cement No. 1						
Repeatability	...	...	0.0072	0.0040	0.0090	0.0063
Reproducibility	...	...	0.0122	0.0110	0.0060	0.0108
Cement No. 2						
Repeatability	...	0.0047	0.0071	0.0059	0.0066	...
Reproducibility	...	0.0074	0.0028	0.0047	0.0119	...
Cement No. 3						
Repeatability	0.0112	0.0191	0.0024	0.0153	...	...
Reproducibility	0.0039	0.0278	0.0050	0.0081	...	...
Cement No. 4						
Repeatability	0.0046	0.0048	0.0039	0.0096	...	...
Reproducibility	0.0075	0.0055	0.0040	0.0044	...	...

Average repeatability	0.0074
Average reproducibility	0.0085
Average repeatability when SO_3 content is at approximate optimum	0.0055
Average reproducibility when SO_3 content is at approximate optimum	0.0046

Let it be assumed that, by virtue of continuing tests, the cement manufacturer is able to determine the optimum SO_3 content of his cement with relatively high accuracy. Let it be further assumed that he is able to evaluate the effects of variations in chemical composition and fineness on optimum SO_3 content and that knowing these results he is able to proportion clinker and gypsum in his mill to result in a variation in SO_3 not in excess of ±0.25 per cent relative to optimum.

If such control were maintained by the manufacturer, it becomes of interest to determine the values of specification limits that could be used to assure the purchaser, as a result of his tests, that the SO_3 content of the cement is within acceptable limits of its optimum value.

For the four cements investigated in this study, it can be determined from the data shown in Fig. 1 that when the SO_3 content is at optimum minus 0.25 per cent, the true change in contraction is virtually nil. Based on the precision of the contraction test, a suitable value of minmium change in contraction is −0.0025 per cent.

For the cements tested, such an observed result in the contraction test with respect to the apparent relationship to optimum is shown in the following tabulation:

Cement...	No. 1	No. 2	No. 3	No. 4
Apparent SO_3 content	+1.65	+1.15	+0.63	−0.05
True optimum SO_3	+2.15	+1.55	+0.98	+0.40
Apparent departure from optimum	−0.50	−0.40	−0.35	−0.45

These results indicate that the purchaser could be assured that the SO_3 content was not deficient by more than about 0.5 per cent relative to optimum.

If the cement were furnished with an SO_3 content of optimum plus 0.25 per cent, the true changes in contraction in the four cements covered in this study are:

Cement No. 1	+0.0053 per cent
Cement No. 2	+0.0077 per cent
Cement No. 3	+0.0080 per cent
Cement No. 4	+0.0063 per cent

These values have been scaled from carefully drawn curves similar to those of Fig. 1.

A suitable specification maximum for change in contraction applicable to these four cements would be 0.0080 + 0.0023 (one half the value of reproducibility) or 0.010. For the cements tested such an observed result in the contraction test with respect to the apparent relationship to optimum is shown in the following tabulation:

Cement...	No. 1	No. 2	No. 3	No. 4
Apparent SO_3 content	+2.85	+2.00	+1.48	+1.00
True optimum SO_3	+2.15	+1.55	+0.98	+0.40
Apparent departure from optimum	+0.70	+0.45	+0.50	+0.60

These results indicate that the purchaser could be assured that the SO_3 content was not in excess of optimum by more than 0.75 per cent. We are however interested in the possibility that the expansion test is able to afford better assurance to the purchaser. A study of the *changes* in expansion produced by 0.5 per cent additions of SO_3 does not indicate that the desired control can be obtained in this manner.

"True" values of expansion in per cent, scaled from Fig. 5, are as follows:

Cement	At optimum +0.25 per cent SO_3	At optimum +0.75 per cent SO_3
No. 1	0.0074	0.0130
No. 2	0.0065	0.0100
No. 3	0.0045	0.0090
No. 4	0.0065	0.0095

For cement produced with an SO_3 content of optimum +0.25 per cent, a suitable specification limit on expansion would be 0.0075 plus 0.0025 (one half the value of reproducibility) or 0.010. For the cements tested, such an observed result in expansion with respect to the apparent relationship to optimum SO_3 content is shown by the following tabulation:

Cement...	No. 1	No. 2	No. 3	No. 4
Apparent SO_3 content	2.75	2.30	1.80	1.25
True optimum SO_3	2.15	1.55	0.98	0.40
Apparent departure from optimum	0.60	0.75	0.82	0.85

The expansion test made on the original cement does not provide as great a degree of assurance to the purchaser as does the contraction test.

When gypsum is added to increase the SO_3 content 0.50 per cent in the cement containing optimum +0.25 per cent SO_3, the cement tested is at optimum +0.75 per cent. A suitable specification limit for this expansion test is 0.010 plus 0.004 (one half the reproducibility) or 0.014 per cent. (The use of this value, in effect, would require that the manufacturer control cement No. 1 below optimum +0.10 per cent).

For the cements tested such an observed result in expansion with respect to the apparent relationship to optimum SO_3 content is shown in the following tabulation:

Cement...	No. 1	No. 2	No. 3	No. 4
Apparent SO_3 content........	2.95	2.60	1.93	2.00
Apparent SO_3 (original cement).........	2.45	2.10	1.43	1.50
True optimum SO_3...........	2.15	1.55	0.98	0.40
Apparent departure from optimum.........	0.30	0.55	0.45	1.10

These results do not indicate that the expansion test of itself affords greater control than that obtainable by the contraction test (except in the case of cement No. 1, for which the selected limit of expansion was more restrictive than for the other cements). Nevertheless, the expansion test can be made with little additional effort, and it is believed that it can serve the purpose of furnishing added assurance to the purchaser.

Precision of Test for Calcium Sulfate in Hydrated Mortar:

Tests for calcium sulfate in hydrated mortar at three progressive additions of SO_3 were made by three laboratories. The criterion has been suggested that optimum SO_3 content lies within the range of more than 0.2 g of SO_3 per liter at 18 hr and less than 0.5 g at 24 hr. The SO_3 content of each cement that results in each of these values has been computed by straight-line interpolation and the results are shown in Table XII. The mean of this range is taken as the optimum SO_3 by this method of test. The range in increments of SO_3 for cement No. 2 did not embrace 0.5 g of SO_3 per liter at 24 hr and therefore the optimum for this cement cannot be computed exactly. For the remaining cements, the data indicate that the three laboratories making this test were able to obtain values of optimum SO_3 within 0.3 per cent of the value established by the cooperative expansion-contraction tests. The results for cement No. 2 departed from the expansion-contraction value by more than 0.55 per cent.

TABLE XII.—INTERPOLATED VALUES OF CALCIUM SULFATE IN HYDRATED MORTAR.

Values are in per cent of SO_3 in excess of that in the original cement required to yield 0.2 g. of SO_3 at 18 hr and 0.5 g at 24 hr.

Cement	Laboratory	0.2 g 18 hr	0.5 g 24 hr	Mean	Optimum by Contraction
No. 1....	No. 1...	2.02	2.42	2.22	
	No. 2...	2.12	2.50	2.31	
	No. 3...	2.10	2.68	2.39	
	Avg...	2.08	2.53	2.31	2.15
No. 2....	No. 1...	1.80	2.50+	2.15+	
	No. 2...	1.75	2.50+	2.13+	
	No. 3...	1.68	2.50+	2.09+	
	Avg...	1.74	2.50+	2.12+	1.55
No. 3....	No. 1...	0.20	1.20	0.70	
	No. 2...	0.20	1.15	0.68	
	No. 3...	0.40	1.24	0.84	
	Avg...	0.27	1.20	0.74	0.98
No. 4....	No. 1...	0.22	1.04	0.63	
	No. 2...	0.12	0.68	0.39	
	No. 3...	0.20	1.05	0.63	
	Avg...	0.18	0.92	0.55	0.40

DRYING CABINET

In the contraction test, four of the laboratories used drying cabinets constructed in accordance with the details shown in the Appendix. The drying conditions within these cabinets varied somewhat between laboratories. In general, the relative humidity rose to about 75 per cent when specimens were first placed in the cabinet. During the succeeding 14 to 18 hr the relative humidity dropped to values ranging between 45 and 60 per cent. One laboratory conducted the contraction test in a room maintained at 73 ± 2 F and 50 to 57 per cent relative humidity.

Notwithstanding the apparent differences in drying conditions among the several laboratories, the data indicate that the magnitude of contraction of similar specimens was reasonably constant. A summary of results is given in Table XIII. The test procedure required that specimens of the same cement with varying additions of SO_3 be molded in close succession on the same day. The differences in contraction for varying additions of SO_3 were more nearly constant among laboratories than were the absolute values. It is concluded that the control of temperature and relative humidity provided by the cabinets was satisfactory for the purpose of the test.

TABLE XIII.—ABSOLUTE VALUES OF CONTRACTION IN PER CENT FOR CEMENTS CONTAINING THE PROGRESSIVE ADDITIONS OF SO_3 NEAREST TO THE OPTIMUM VALUE.

Laboratory	Cement				
	No. 1	No. 2	No. 3	No. 4	Average
No. 1.....	0.0553	0.0582	0.0435	0.0332	0.0462
No. 2.....	0.0611	0.0551	0.0439	0.0350	0.0488
No. 3.....	0.0598	0.0546	0.0437	0.0354	0.0484
No. 4.....	0.0552	0.0504	0.0396	0.0286	0.0440
No. 5.....	0.0578	0.0483	0.0432	0.0337	0.0463
Average...	0.0573	0.0533	0.0428	0.0352	0.0470

COMPUTATION OF CEMENT-GYPSUM BLENDS

Computation of the amount of gypsum to be blended with the original cement to obtain the desired increment of SO_3 depends on the SO_3 content of the original cement. The error introduced by assuming a fixed SO_3 content in the original cement, say 2.0 per cent, is not great. Routine test procedure can be simplified by such an assumption to eliminate the necessity of determining SO_3. If this is done, the formula for computing added gypsum to produce an increase of 0.5 per cent SO_3 becomes:

$$X = \frac{0.5}{C - 2.00} \times 100$$

$$Y = 100 - X$$

where:

X = per cent of gypsum in the blend,
Y = per cent of cement in the blend, and
C = SO_3 content of the gypsum.

ABSOLUTE VALUES OF OPTIMUM SO_3

Throughout this discussion, the SO_3 content of the cements and blends has been expressed in terms of the progressive additions relative to the original cement. It is of interest to consider the absolute values of optimum SO_3 and these are given below:

Cement No. 1, type III, high C_3A, low alkali....................	3.86 per cent
Cement No. 2, type III, low C_3A, low alkali....................	3.04 per cent
Cement No. 3, type I, high C_3A, high alkali....................	3.26 per cent
Cement No. 4, Type II, low C_3A, high alkali....................	2.10 per cent

DURATION OF DRYING

Laboratory No. 1 submitted data of contraction of the four cements at the ages of 7, 14, 21 and 28 days, during which the specimens were subjected to drying for 4, 11, 18 and 25 days. The data when plotted as change of rate in contraction curves, similar to those shown in Fig. 1, indicated optimum SO_3 contents as shown in Table XIV.

The data indicate a gradual increase in the amount of SO_3 required for optimum as the period of drying is increased. Optimum SO_3 after 25 days of drying is about 0.20 per cent higher than that after 4 days of drying.

CONCLUSIONS

The results of these cooperative tests on four cements, to the extent that they are applicable to all cements, indicate the following with respect to single tests made upon the cement as received and upon the same cement to which gypsum has been added to increase the SO_3 content by 0.5 percentage point.

The contraction test can provide assurance to the purchaser that the SO_3 content lies within the range of −0.50 per cent to +0.75 per cent relative to optimum. To provide such assurance it is necessary that the manufacturer control the SO_3 content within ±0.25 percentage point of the optimum. Suitable specification limits to provide such assurance to the purchaser are that the difference in contraction shall not be less than −0.0025 nor greater than +0.010 per cent.

The expansion test, while not more discriminating than the contraction test with respect to an excess of SO_3, affords additional assurance against such an excess. Suitable specification limits for expansion of the original cement are not more than 0.010 per cent, and, for the cement to which gypsum has been added to increase the SO_3 content by 0.5 per cent, not more than 0.014 per cent.

Although the relative precision of the compressive strength test is shown in this study to be less than that of the expansion-contraction test, the fact that test results can be obtained within 24 hr suggests the desirability of further investigation. Although the three laboratories that performed this test for calcium sulfate in hydrated cement mortar (modified to obtain results at 18 and 24 hr) were able to obtain the same order of precision as in the expansion-contraction test, the data are not sufficiently extensive to warrant the conclusion that it possesses the same degree of merit.

The expansion-contraction test with limits open enough to apply to all cements does not appear to afford as high a degree of control as desirable. If suitable test limits are applied to individual cements, or to individual types of cement, it appears possible to use the test to obtain reasonably good control from the standpoint of the consumer as well as the producer. Programs of cooperative testing between producer and consumer of single types of cement produced at individual mills are encouraged as means of determining feasible control limits for the cements under investigation.

TABLE XIV.—ADDITION OF SO_3 REQUIRED FOR OPTIMUM AFTER DRYING FOR THE PERIOD INDICATED.

Cement	4 days	11 days	18 days	25 days
No. 1	2.12	2.22	2.24	2.32
No. 2	1.57	1.68	1.73	1.77
No. 3	0.97	1.05	1.12	1.15
No. 4	0.40	0.55	0.60	0.62

Reported by the working group on SO_3 content,

T. B. KENNEDY, *Chairman*
O. E. BROWN
W. C. HANSEN
WILLIAM LERCH
W. J. MCCOY
J. S. OFFUTT
K. E. PALMER
C. U. PIERSON, JR.
BAILEY TREMPER
J. F. WEIGEL

METHOD OF TEST FOR EXPANSION IN WATER AND CONTRACTION IN AIR OF PORTLAND-CEMENT MORTAR

Scope

1. This method is intended to measure the expansion in water of mortars containing portland cement and a blend of the cement and gypsum. It is also intended to measure the change in contraction in air of mortar produced by the addition of gypsum to the portland cement. The results may be used to indicate the relationship of the SO_3 content of the cement to its optimum content.

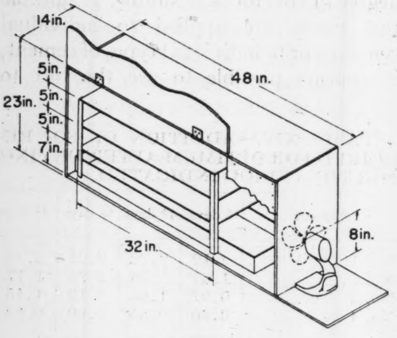


FIG. 6.—Drying Cabinet.

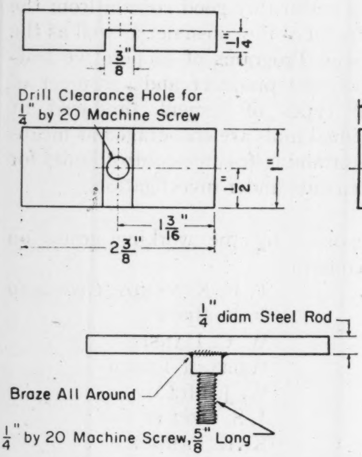

3/8"
1/4"
Drill Clearance Hole for 1/4" by 20 Machine Screw
1/2"
1"
1/2"
1 3/16"
2 3/8"
1/4" diam Steel Rod
Braze All Around
1/4" by 20 Machine Screw, 5/8" Long

FIG. 7.—Device to Facilitate Removal of Molded Specimens.

Apparatus

2. Apparatus specified in Method C 109,[1] less specimen molds and testing machine.

(*a*) *Molds and comparator*, as specified in Method C 151.[2]

(*b*) *Drying cabinet*, constructed according to the design shown in Fig. 6. The pan shall contain a saturated solution of sodium dichromate with an excess of crystals. The depth of solution in the pan shall be about 1½ in. Spread the sodium dichromate crystals over the bottom of the pan to a loose depth of about 1 in. (about 10 lb required). Add water to bring the depth to approximately 1½ in. Stir to effect substantial saturation. Add additional sodium dichromate if necessary to produce or maintain an excess of undissolved salt.

The cabinet shall be installed in a room, the temperature of which is maintained between 68 and 81.5 F.

In lieu of the above cabinet, any enclosure equipped with suitable shelves or racks may be used provided the temperature within the enclosure is maintained between 68 and 81.5 F, and the relative humidity is maintained between 45 and 60 per cent.

(*c*) *Device*, as detailed in Fig. 7 to facilitate removal of molded specimens from the base plate.

Materials

3. (*a*) *Graded Ottawa sand*, see Section 4, Method C 109.

(*b*) *Sodium dichromate* ($Na_2Cr_2O_7$), crystals, technical grade.

(*c*) *Finely pulverized gypsum*, of the grade known commercially as Terra Alba, containing not less than 40 per cent SO_3. At least 90 per cent shall pass a No. 325 sieve.[3]

---

[1] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens (C 109 – 58), 1958 Book of ASTM Standards, Part 4.

[2] Methods of Test for Autoclave Expansion of Portland Cement (C 151 – 58), 1958 Book of ASTM Standards, Part 4.

(*d*) *Masking tape,* 1 in. wide.

Procedure

4. (*a*) A batch of mortar shall consist of 750 g cement or cement-gypsum blend, 1500 g graded Ottawa sand and water in the quantity required to produce a flow of the straight cement mortar between 100 and 115 when tested in accordance with Method C 109. The same amount of water shall be used in the mortar containing the cement-gypsum blend.

(*b*) Mix one batch of mortar with the cement under test. Mix a second batch within 30 min with the cement blended with pulverized gypsum in an amount necessary to increase the SO_3 content approximately 0.50 percentage points. Calculate the weights of cement and gypsum required to produce the required blend as follows:

$$X = \frac{0.5}{C - 2.00} \times 750$$

$$Y = 750 - X$$

where:

X = g gypsum in the blend,
Y = g cement in the blend, and
C = SO_3 content of the gypsum in per cent.

(*c*) Prepare the cement-gypsum blend in lots of the size required for a single batch of mortar. Weigh the gypsum to the nearest 0.1 g using a suitable balance. Add cement to bring the total weight of the blend to 750 g. Transfer the weighed mixture to a bowl and mix with a rubber spatula until the cement and gypsum appear to be uniformly blended. If the blend is not used at once for mixing mortar, transfer it to a tight container.

(*d*) Mix the mortar according to Method C 109.

(*e*) Mold four 1 by 1 by 10-in. gage length bars from each batch of mortar as specified in Section 4(*c*), Method C 151.

(*f*) Store the specimens in the moist closet for 24 hr ± 30 min, computed from the time of mixing. Remove the specimens promptly from the molds. (The use of the device illustrated in Fig. 7 is recommended as a precaution against breakage of the bars.) Measure the length as provided in Method C 151. If not measured at once, protect the specimens by covering them with a damp cloth.

(*g*) Store the specimens in water maintained at a temperature of 73.4 ± 3 F to the age of 72 hr ± 30 min, computed from the time of mixing. Remove the specimens from the water, wipe free of surface moisture and measure promptly for length.

(*h*) Place the specimens on shelves of drying cabinet with at least 1 in. clear space between specimens. Close the door of the cabinet and seal with masking tape. Start the fan. Leave the door closed and operate the fan continuously except when adding or removing specimens. Remove specimens when they have reached the age of 168 hr (7 days) ± 30 min, computed from the time of mixing. Measure promptly for length.

Calculation

5. (*a*) Calculate the expansion in water of each specimen as the length upon removal from water storage less the length upon removal from the molds and express it as a percentage of 10 in.

(*b*) Calculate the contraction in air of each specimen as the length upon removal from water storage less the length upon removal from the drying cabinet and express it as a percentage of 10 in.

(*c*) The average change in length of four specimens from the same batch of mortar shall be reported as the per cent expansion in water or the per cent contraction in air provided the variability between specimens does not exceed the limits of acceptability as described below. If the results of four specimens do not meet the criterion for acceptability, but the results of any three of the specimens do meet the criterion, the average of the measured length changes of the three specimens shall be reported as the per cent expansion in water or the per cent contraction in air.

---

[3] Tentative Specifications for Sieves for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Screens or Sieves) (E 11), 1958 Book of ASTM Standards, Part 4.

(*d*) If the length changes of at least three specimens do not meet the criterion for acceptability, the results shall not be reported and the test shall be repeated.

(*e*) The criterion of acceptability of test results shall be that the standard deviation of the measured length changes of the number of specimens considered does not exceed 0.0025 in expansion nor 0.0030 in contraction. Compute the standard deviation as follows:

$$\sigma = \sqrt{\frac{\Sigma(X - \bar{X})^2}{n - 1}}$$

where:

σ = the standard deviation,
X = a single observation,
$\bar{X}$ = the arithmetic mean of all observations under consideration, and
n = the number of observations under consideration.

Report

6. The report of the test shall show the per cent expansion in water and the per cent contraction in air of the original cement and the cement-gypsum blend, the standard deviation for each value and the number of specimens considered in computing the average value. The report shall also show the difference between the per cent contraction in air of the original cement and the cement-gypsum blend. This value shall be prefixed with a minus sign if the contraction of the original cement is greater than that of the cement-gypsum blend.

APPENDIX II

THE CEMENT REFERENCE LABORATORY*
(1929–1959)

BY J. R. DISE[1]

SYNOPSIS

Recent changes in the scope of its operations have stimulated numerous inquiries about the functions of the Cement Reference Laboratory. Therefore, as a record which may be of special interest to those engaged in the testing of cements and concretes, and of general interest to those engaged in the testing of other materials, a report has been prepared which covers the origin, purpose, and development of each of the present responsibilities of the Cement Reference Laboratory.

The work of the Cement Reference Laboratory (CRL) in the cement and concrete testing fields has been of interest to many groups concerned with the development and improvement of methods of testing materials. In consideration of this interest, it seems very appropriate on the occasion of its thirtieth anniversary to present a report on the origin and development of the various services rendered by this organization.

EVENTS LEADING TO THE ESTABLISHMENT OF THE CRL

In the early part of the century, various technical organizations, including the National Bureau of Standards, the U. S. Army Corps of Engineers, the American Society of Civil Engineers, Committee C-1 on Cement of the American Society for Testing Materials, and the Portland Cement Assn., became actively engaged in efforts to improve and standardize the specifications and methods of test for portland cement. As this work progressed, numerous cooperative investigations were undertaken by groups of laboratories. In general, the concordance of test results was not satisfactory. In 1926, following an extensive series of cooperative tests, it was reported to ASTM Committee C-1 that "the variations among the results of the participating laboratories were so great that any definite recommendations as to the relative merits of test specimens or methods appeared unlikely until some means of training would secure greater uniformity in the operators' work (**1**)."[2] When later cooperative tests only served to emphasize the problem, Committee C-1 appointed, in 1928, a Special Committee on Reference Laboratory to bring about the establishment of a laboratory devoted to the promotion of uniformity in the testing of cement.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] In charge, Cement Reference Laboratory, National Bureau of Standards, Washington, D. C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

SPONSORSHIP

As a result of the Special Committee's work, the Cement Reference Laboratory was established in April, 1929, as a Research Fellowship at the National Bureau of Standards with ASTM Committee C-1 as the sponsor of the Fellowship. Committee C-1 appointed the subcommittee on the Cement Reference Laboratory as its representative, and subsequently the National Bureau of Standards designated the Concreting Materials Section as its representative in the supervision of the enterprise. Representatives of the U. S. Bureau of Public Roads and the U. S. Army Corps of Engineers, who later joined the original sponsors in support of the operation, have very actively participated in the administration for many years through membership on the CRL Subcommittee.

TABLE I.—CONFORMANCE OF APPARATUS EXAMINED TO SPECIFICATION REQUIREMENTS.[a]

	Tour 1	Tour 2	Tour 3	Tour 4[b]
	Conforming, per cent	Conforming, per cent	Conforming, per cent	Conforming per cent
Steam chests	48	63	78	92
Balances for mixes	74	68	86	89
Balances for fineness	33	49	76	92
Tensile machines	43	62	80	87
Vicat apparatus	30	51	80	82
Gillmore needles	58	61	89	95
Briquet molds	67	80	92	92
Graduates	84	92	94	93
Weights for mixes	97	93	98	99.7
Weights for fineness	95	95	98	99.6
Approximate tour period	1929–1930	1931–1932	1932–1933	1934–1935

[a] Data obtained from *Proceedings*, Am. Soc. Testing Mats., Vol. 35, p. 237 (1935).

[b] Data covering the 143 inspections made in tour 4 prior to preparation of this table.

RESPONSIBILITIES

All four of the operations which were to become the major functions of the Reference Laboratory were enumerated in the initial plans for the project. These functions, and the various changes that have been made in their scope since 1929, are described in the following sections of this report:

The Laboratory Inspection Services
Studies of Apparatus and Methods
The Distribution of Comparative Test Samples
Participation in the Work of Technical Committees

THE LABORATORY INSPECTION SERVICES

The inspection of cement testing laboratories was indicated as the primary function of the Reference Laboratory in the original plans because it seemed that uniformity in apparatus and methods might best be promoted through educational work in the field. Later, when it became evident that the desired results were being obtained (see Table I), it was concluded that the development of new methods of test, frequent changes in testing laboratory personnel, and the establishment of new laboratories required continuation of the inspection work on a permanent basis.

During the first eighteen years, inspection coverage was restricted to tests used in determining the physical properties of hydraulic cements. In 1948, the scope was broadened by undertaking the verification of large-capacity compression testing machines used in the testing of concrete by the cement laboratories which were then receiving inspections.

Subsequently, this work was expanded to include inspection of most of the methods and equipment used in the determination of compressive strength of concrete. On March 1, 1958, the new inspection service was made available to any concrete testing laboratory within the areas normally covered by the CRL Inspectors.

General Policies:

Inspections are limited to laboratories making the standard tests of cement or concrete. Utilization of the services is on a voluntary basis, and inspections are made only as requested by interested laboratories. Inspection itineraries are arranged so that these laboratories may be visited at intervals of from two to three years. The work is advisory in nature, and there is no "police-type" action involved. The Reference Laboratory is not permitted to assume any legal responsibilities, and therefore no efforts are made to certify or rate laboratories in any manner or to serve as a referee in disputes relating to quality of materials or test equipment.

The Nature of the Services:

The inspection services consist of the examination of testing apparatus, and instructions in methods of test.

In general, the examination of apparatus is limited to equipment required by the current ASTM physical tests for cement and tests for compressive strength of concrete. In some few instances, additional work is undertaken to develop information that may be of particular interest to the CRL or its sponsors. Reports on the inspection of apparatus mention whether it meets, or fails to meet specification requirements, and whether or not it was in satisfactory operating condition. No statements of "rejection" are made. Where feasible, each piece of satisfactory apparatus is assigned a permanent CRL identification number at the time of its first inspection. Virtually all of this work is done in the course of visits to laboratories and, as a matter of policy, no equipment is examined while it is still in the hands of the manufacturer.

Each inspector in the field is equipped with approximately 800 lb of equipment which has been carefully selected and calibrated for use in the checking of testing apparatus. The elaborate kit includes precision weights; a small balance; various gages, thermometers, and psychrometers; a small test lever; four proving rings of various capacities up to 200,000 lb, a hydraulic gage tester; a microammeter test set; and a considerable assortment of micrometers, scales, straightedges, small tools, and reference literature. These items are transported in panel type trucks, most of which are specially modified for this usage. For comparison, it is noted that in 1929 an inspector traveled by train and carried his few items of test equipment in one medium-size trunk and two small boxes.

Instruction in methods of test has been largely limited to the standard ASTM methods for determining the physical properties of cements. Under current practices, the inspector observes a demonstration of the standard methods by a laboratory's physical tester and offers comments on the details which may require correction or merit discussion. In instances where wide departures are noted, the inspector demonstrates the standard methods. If improper usage or care of testing apparatus has been noted, he also comments on these matters. It is estimated that at least 2000 laboratory technicians have received instruction from the CRL inspectors. This figure seems conservative because in tour 10 alone, 243 of the 347 participating physical testers had not participated in tour 9.

Reports on Inspections:

On completion of his work in a laboratory, the CRL representative confers with an appropriate member of the laboratory staff, making available to that individual the data on the inspector's work sheets, and discussing all important observations which were made during the course of the visit. At an early date thereafter, a detailed confirmatory report, containing an informal document which may be displayed in the laboratory as an evidence of the inspection, is sent to the official who requested the visit. Also, in many instances, copies of the report are forwarded to other interested officials of the inspected laboratory.

TABLE II.—CLASSIFICATION AND NUMBER OF LABORATORIES PARTICIPATING IN VARIOUS INSPECTION TOURS.

Laboratory Classification	Tour 1	Tour 10		Tour 11		Tour 12[a]	
	Cement Testing	Cement Testing	Concrete Testing	Cement Testing	Concrete Testing	Cement Testing	Concrete Testing
Cement producer	99	140	22	165	20	150	15
Commercial testing	45	39	38	24	24	23	43
Highway Department	25	48	43	39	37	41	40
School	21	9	8	4	3	1	2
Municipal	11	4	4	3	1	3	1
Federal	3	13	9	12	8	14	11
Miscellaneous	4	8	7	6	5	8	8
Total inspections	208	261	131	253	98	240	120
Number of participating laboratories	208	261		253		265	
Number of new participants		34		15		27	
Approximate tour period	1929–1930	1950–1954		1955–1957		1957–1960	

[a] Estimate of participants based on requests received prior to Jan. 1 1959.

The inspector's findings, and the confirmatory report, are regarded as confidential by the CRL staff. However, it is possible for anyone who applies to the CRL to obtain a copy of an inspection report which might be of particular interest when the laboratory concerned is agreeable to such distribution. Since inspected laboratories are not authorized to reproduce a report, or any part thereof, for use in advertising, publication, or promotional work, the distribution must be made through the National Bureau of Standards. This procedure for obtaining inspection information has been used extensively by the Bureau of Public Roads in the appraisal of testing laboratories engaged in work related to Federal Aid Projects.

Number and Interests of Inspected Laboratories:

As of Jan. 1, 1959, 2903 inspections had been performed in the more than 520 laboratories that have availed themselves of the inspection services during this 30-yr interval. The laboratories have been widely scattered, and the work has taken inspectors into 48 states of the United States, 6 provinces of Canada, and Puerto Rico.

Information concerning the number of laboratories participating in some of the recent inspection tours of the areas in which the CRL operates and the classifications of these laboratories according to the interests represented is given in Table II. Data from tour 1 has been included for comparative purposes. Similar information for tours 2 to 9 may be found in the "History of the Cement Reference Laboratory 1929–1955 (2)." In Table II, the tabulation of inspections performed in concrete testing laboratories constitutes a progress report on this phase of the inspection work.

It is to be observed that developments in recent years have validated the reasons for giving permanent status to the inspection work among cement testing laboratories. The development of new methods and specifications may be illustrated by the fact that there are now 33 ASTM standards relating to cement, whereas there were only 21 such standards in 1946. The frequent changes in testing laboratory personnel is evidenced by the 70 per cent turn-over in physical testers, participating in the inspections, that occurred between tours 9 and 10. The establishment of new laboratories is indicated by the fact that approximately 10 per cent of those participating in each new tour have not been inspected in any of the previous tours. Because cement testing laboratories and concrete testing laboratories have much in common, these developments in the cement testing field became important factors in the deliberations relative to extension of the inspection services in the concrete testing field.

STUDIES OF APPARATUS AND METHODS

The initial plans stated that, as time and means permitted, the Reference Laboratory would make studies of methods, apparatus, and conditions which might be useful to Committee C-1 in its efforts to improve concordance of results in cement testing. Accordingly, as opportunities have arisen, the staff has undertaken both laboratory and field studies of various problems which seemed to require attention and has participated in numerous cooperative studies conducted by the Committee C-1 Working Committees and other technical groups.

Special Studies:

Since the inspection work is more closely related to the practical than to the theoretical aspects of testing, the studies have usually dealt with topical problems of a practical nature. A review of the various projects completed between 1929 and 1955 (a listing is appended to the 1957 Annual Report of Committee C-1 (2)) indicates that the principal areas of interest have been methods for determining fineness of portland cement; tests of additions to portland cement; and methods of test for determining the compressive strength and the air content of mortars. Probably the best known projects are L. A. Wagner's work (3) on the turbidimetric method for determination of the fineness of portland cement and the development of the CRL flow table mixture for checking the performance of the 10-in. flow tables used in tests for consistency of hydraulic cement mortars.

The development of the flow calibration material was typical of the combined laboratory-field investigations that have been conducted over the years. Work on this project was undertaken to satisfy the desire of the CRL inspectors for a material that could be used for checking flow table performance in the field. Mixtures used by earlier investigators were tried and found to be unsuited to the inspection work. Later, an assortment of mixtures was prepared by adding various ground materials to oil. From these a mixture consisting of 500 g of silica flour, specially ground by the CRL, and 350 g of a selected mineral oil was chosen for field usage. Gradually, through experimentation by the CRL staff, and others, several important difficulties encountered in the early field trials were eliminated, and this mixture is now established as the calibration material for flow tables.

Coincidentally with the development work, the preparation and distribution of the flow table material became one of the responsibilities of the CRL. Since 1947, when the first experiments were conducted, more than 750 cartons each containing the ingredients for one sample have been forwarded to laboratories in the United States, Canada, Puerto Rico, New Zealand, South America, and Europe. Suggestions for improvement of flow table performance, based on CRL investigations, have accompanied these samples.

TABLE III.—FLOW TABLE TESTS MADE WITH SAMPLES FROM LOT F OF THE CEMENT REFERENCE LABORATORY FLOW TABLE MATERIAL.[a]

Laboratory	Temperature of Material, deg Fahr	Percentage Flow After 10 Drops					Percentage Flow After 25 Drops[b]				
		Table Top Position—Rotated from Normal, deg					Table Top Position—Rotated from Normal, deg				
		Normal	90	180	270	Average	Normal	90	180	270	Average
No. 1........	73	79.5	79.8	79.5	80.3	79.8	103.5	103.3	104.0	103.8	103.7
No. 2........	76	76.8	77.0	77.3	76.0	76.8	103.1	102.5	102.8	101.1	102.4
No. 3........	74	80.0	81.0	80.0	81.0	80.5	106.0	106.0	106.5	106.5	106.3
No. 4........	75	76.8	76.5	77.3	77.5	77.0	100.0	100.5	100.8	100.9	100.6
No. 5........	78	80.8	80.8	80.8	80.8	80.8	104.5	104.8	105.0	104.8	104.8
No. 6........	75	78.8	78.5	80.0	78.0	78.8	101.3	102.0	102.8	100.3	101.6
No. 7........	76	84.3	84.4	84.5	84.7	84.5	107.8	109.0	109.4	109.3	108.9
No. 8........	79	83.8	85.1	85.6	85.1	84.9	107.2	107.2	108.5	109.2	108.0
No. 9........	77	82.0	81.5	81.8	80.8	81.5	105.8	106.5	106.3	106.3	106.2
No. 10........	73	77.5	77.3	77.3	77.8	77.5	102.3	102.0	102.0	101.8	102.0
No. 11........	74	78.8	79.0	78.8	78.8	78.9	103.0	103.0	103.2	103.2	103.1
No. 12........	76	77.1	77.4	77.3	77.3	77.3	101.5	101.3	101.7	101.1	101.4
No. 13........	76	75.0	75.6	75.4	75.9	75.5	98.1	99.6	98.3	98.9	98.7

[a] Coordinated check tests made in Dec., 1956 by 13 different laboratories in one organization, with each laboratory using a separate sample of the flow material.

[b] Performance of a flow table is considered to be satisfactory if, in calibration tests with a suitable material, the table gives a flow value that does not differ by more than 5 percentage points from the flow value assigned to the calibration material. Lot F of the CRL flow table material had an assigned flow value of 103 per cent.

An example of the use of the material for comparison of flow table performance is given in Table III. These data have been contributed by one of the organizations that cooperated in the development work.

Cooperative Work:

In April 1929, the Reference Laboratory was asked to be prepared to make recommendations for certain tolerances for testing apparatus to the Subcommittee on Apparatus of ASTM Committee C-1 by June 1929. This was the first cooperative study in which the CRL participated. Later in the year, the laboratory collaborated in some special sieve tests of certain lots of standard No. 20 to No. 30 sand conducted by C-1 Subcommittee III on Fineness. Several of the subsequent recommendations of the subcommittees, based on these studies, are still in effect.

The activities described above are typical of the cooperative projects in which the staff became engaged as the CRL continued its work. Information on the more important of the past undertakings is contained in the "History of the Cement Reference Laboratory (2)." Current cooperative projects to which the CRL has contributed include the development of procedures for the mechanical mixing of cement pastes, the study of specifications for molds for concrete test cylinders, and the development of a "Manual on Concrete Testing."

DISTRIBUTION OF COMPARATIVE TEST SAMPLES

The distribution of comparative test samples of cement to laboratories who expressed a desire to make such tests was listed in the original plans as a service that might be rendered by the Reference Laboratory. The scope of such work was to be governed by future demands for the service.

TABLE IV.—IMPROVEMENT IN REPRODUCIBILITY OF RESULTS FOR TESTS FOR AIR CONTENT OF MORTAR.

Sample number	6[a]	9[b]	12[b]
Year distributed	1954	1955	1957
Number of results	179	183	211
Departure from Average Air Content, per cent	Participants Within the Indicated Range, per cent		
Within ±1	35.4	72.7	80.5
Over 1.0 under 2.0	42.1	19.1	15.2
Over 2.0 under 3.0	12.4	4.4	2.4
Over 3.0	10.1	3.8	1.9

[a] Mortars mixed by hand.

[b] Mortars mixed with mechanical mixer.

The first venture in this field was the experimental distribution of a cement sample to 45 testing laboratories in 1936. This work was followed in 1938 by the distribution of a sample to all interested laboratories. Twelve other samples have been distributed in subsequent years. The interest that has been shown in this program indicates that laboratories welcome the opportunity to engage in comparative testing.

Much of the work related to the 14 samples has been carried out in cooperation with working groups within Committee C-1. A summation of the program shows that portland cements, air-entraining portland cements, high-early strength portland cements, and a masonry cement have been tested, and that chemical tests have been conducted as frequently as physical tests. In many instances, the collection of special information about test procedures and details of apparatus and testing conditions were made a part of the work. For these reasons, the data and related notes and correspondence have generally been of interest to subcommittees of ASTM Committee C-1, and others, engaged in the development of specifications and methods of test.

The data from the comparative tests have provided the CRL with a helpful means for checking on the general status of progress toward better concordance in test results and on the possible contribution of the inspection service to such progress. This usage of the data is illustrated by the information in Table IV which shows the improvement in reproducibility of results occurring from changes in the method for determining the air content of mortar, including adoption of mechanical mixing of the mortar, and the continuation of this improvement as closer conformance to the new procedures was promoted through the inspection work.

PARTICIPATION IN THE WORK OF TECHNICAL COMMITTEES

The early plans for CRL participation in the work of technical committees consisted simply of an understanding that any findings would be reported to the appropriate subcommittees of Committee C-1. The scope of this activity has gradually increased to the point where committee work constitutes a major function of the Reference Laboratory.

Since 1931, the CRL Supervisor has served as the Technical Assistant to Committee C-1 on Cement. The principal duty of this appointed officer of the committee is to handle its routine business affairs. Other responsibilities include chairmanship of the Working Committee on Coordination of Methods of Test, and membership on the Working Committee on Methods of Test for Air-Entrainment. As a representative of Committee C-1 to ASTM Committee E-1 on Methods of Testing, the Technical Assistant has been made a member of the E-1 Subcommittees on Calibration of Mechanical Testing Machines and Apparatus, Effect of Speed in Mechanical Testing, Conditioning and Weathering, Ceramic Laboratory Apparatus, and Metalware Laboratory Apparatus. Also, in recent years, a member of the CRL staff has represented Committee C-1 on ASTM Committee C-9 on Concrete and Concrete Aggregates.

ADMINISTRATIVE INFORMATION

The individual and organizational support provided the Reference Laboratory is essential to its welfare. The principal supporting organizations are the National Bureau of Standards, the American Society for Testing Materials, the U. S. Army Corps of Engineers, the Bureau of Public Roads, and the Portland Cement Assn. Operating procedures are largely determined by the recommendations Committee C-1 on Cement makes to the ASTM and to the National Bureau of Standards upon the advice of its Subcommittee on the Cement Reference Laboratory[3] and by the policies of the National Bureau of Standards. Responsibility for the coordination of these supporting efforts rests with the Chief of the Concreting Materials Section of the National Bureau of Standards (R. L. Blaine); the Chairman of Committee C-1 (R. R. Litehiser); and the Chairman of the C-1 Subcommittee on the Cement Reference Laboratory (A. A. Bates).

CONCLUSION

In its report to Committee C-1 on Oct. 19, 1928, the Special Committee on the Cement Reference Laboratory advised "Greater concordance in results is necessary if the requirements of the specifications are to closely reflect the qualities of the product, and if we are to remove a most undesirable uncertainty in both the technical and commercial ends of the cement industry." The progress that has been made toward greater concordance in the results reported by cement testing laboratories during the past 30 years, and the contribution of the Reference Laboratory to this progress were recently described in the ASTM BULLETIN by the statement "the Cement Reference Laboratory (CRL) has been a major factor in bringing about a satisfactory degree of uniformity and voluntary agreement among the producers and consumers of cement and concrete with regard to the methods of testing cements (4)." It has been the purpose of this report to give an accounting of the procedures used by the CRL in carrying out its assignment.

Uniformity in methods and equipment is essential to the continued development of better practices and specifications, and the extensive technological advances occurring in both the cement and concrete testing fields are creating an increasing need for the services of the Reference Laboratory. It is hoped that in the future concrete testing laboratories will derive as much assistance from the CRL services in their field as cement testing laboratories have derived from the long-established services in their field.

Acknowledgments:

Much of the credit for the accomplishments described in this report is attributed to P. H. Bates, J. R. Dwyer, and G. E. Warren for their work in the guidance of the Cement Reference Labora-

---

[3] The present members of the ASTM C-1 Subcomittee on the Cement Reference Laboratory are: Harold Allen, A. A. Bates, R. L. Blaine, C. L. Davis, W. C. Hanna, H. F. Hedderich, R. R. Litehiser, W. H. Price, F. V. Reagel, Bailey Tremper, and Stanton Walker.

tory during its formative years. Acknowledgment is made of the extensive technical assistance received from the staff of the National Bureau of Standards. The contributions of the past and present members of the Subcommittee on the Cement Reference Laboratory of ASTM Committee C-1 are gratefully acknowledged. Further expressions of appreciation are rendered to all those who are listed in the "History of the Cement Reference Laboratory" (2) as having assisted materially in the development of the project.

REFERENCES

(1) J. R. Dwyer, "The Cement Reference Laboratory," ASTM BULLETIN, No. 87, Aug., 1937, p. 12.

(2) "History of the Cement Reference Laboratory 1929–1955," Report of Committee C-1, Appendix I, *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 232 (1957).

(3) L. A. Wagner, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 553 (1933).

(4) "Services of Cement Reference Laboratory Extended to Concrete," ASTM BULLETIN, No. 228, Feb., 1958, p. 16.

REPORT OF COMMITTEE C-3
ON
CHEMICAL-RESISTANT MORTARS*

Committee C-3 on Chemical-Resistant Mortars held two meetings during the year: on September 11 and 12, 1958 in Watertown, N. Y.; and on February 5 and 6, 1959 in Pittsburgh, Pa.

The committee consists of 26 members, of whom 12 are classified as producers, 7 as consumers, 5 as general interest members, and 2 as consulting members.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Method of Test for Chemical Resistance of Mortars as appended hereto.[1] This new tentative is intended to replace the Standard Method of Test for Chemical Resistance of Hydraulic Cement Mortars (C 267 - 54).[2]

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Recommended Practice for Installing Sulfur Mortar Joints (C 386 - 56 T):[2]

New Section.—Add a new Section 7 entitled "Chemical Resistance" to read as follows:

7. *Chemical Resistance.*—Sulfur mortars are resistant to most acids and neutral solutions provided that the service temperature does not exceed 190 F. Sulfur cement mortars are generally found to be particularly advantageous when the service to which they may be subjected involves certain oxidizing agents. They do not have good resistance to alkaline solutions, fats, greases, oils, or to organic solvents in general. They are commonly filled with silica. For hydrofluoric acid service carbon fillers are usually used.

Tentative Recommended Practice for Use of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 397 - 57 T):[2]

Section 2(b).—Change the second sentence to read: "Although not recommended, it is permissible to use liquid binder that has inadvertently been frozen if the liquid is thawed and thoroughly remixed before use, provided it can be restored to its original consistency."

Section 8.—Change to read as follows:

8. *Chemical Resistance.*—Silicate mortars are resistant to most acids (except hydrofluoric and concentrated orthophosphoric acids) of any concentration. These mortars are resistant to water and neutral salt solutions for short periods after prolonged exposure to acids. They are not resistant to alkalies of any concentration, nor to boiling water and steam. Since silicate mortars are porous, crystal formation with subsequent growth within the pores can disintegrate these mortars. In service involving salts or salt solutions, changes in temperature or concentration can cause internal crystal formation. Sulfuric acid under certain conditions can react with the mortar to produce internal crystallization. Where such service conditions are involved, the manufacturer should be consulted. The maximum recommended continuous operating temperature for silica filled sodium silicate cement is approximately 950 F.

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

[1] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

Tentative Recommended Practice for Use of Resin-Type Chemical-Resistant Mortars (C 399 - 57 T):[2]

New Section.—Add a new section entitled "Chemical Resistance" to read as follows:

11. *Chemical Resistance.*—A general guide for chemical resistance of resinous mortars for room temperature service is briefly as follows. The ratings are for immersion service and may usually be upgraded for spillage only. Specific recommendations should be obtained from the manufacturer where conditions are questionable.

	Furan	Phenolic	Epoxy	Polyester
Acids, mineral[a] (nonoxidizing)	R	R	R	R
Acids, mineral (oxidizing)	N	N	N	L
Acids, organic	R	R	L	L
Alkalies, inorganic	R	N	R	N
Bleaches	N	N	N	R
Wet gases, oxidizing	N	N	N	R
Wet gases, reducing	R	R	R	R
Gases, nonoxidizing and nonreducing	R	R	R[b]	R
Organic solvents	R	L	L	N

NOTE.—R = generally recommended; L = limited use; and N = not recommended.

[a] Silica filler should be avoided for hydrofluoric acid service. Sulfuric acid above 85 per cent is considered oxidizing at room temperature.

[b] N for ammonia.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specification for:

Resin-Type Chemical-Resistant Mortars (C 395 - 57 T).

Tentative Method of Test for:

Compressive Strength of Chemically Setting, Silicate-Type Chemical-Resistant Mortars (C 396 - 57 T).

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 26 members; 19 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. R. ALLEN,
Chairman.

E. A. REINECK,
Secretary.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-4
ON
CLAY PIPE*

Committee C-4 on Clay Pipe held one meeting during the year: in Boston, Mass., on June 24, 1958, during the Annual Meeting of the Society.

Revision of Scope.—The Board of Directors of the Society approved the revision in scope of Committee C-4 as recommended by the committee. The scope now reads as follows:

> The formulation of specification, nomenclature, and methods of test for vitrified clay pipe, vitrified clay liner plates, and factory-made compression joints used for the conveyance of sewage, industrial waste, and storm waters; and for clay flue linings.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1958 Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision and Reversion to Tentative of:

Recommended Practice for Installing Clay Sewer Pipe (C 12 – 58).

New Tentative Specification for:

Vitrified Clay Pipe Joints Using Materials Having Resilient Properties (C 425 – 58 T).

These recommendations were accepted by the Standards Committee on September 9, 1958. The revised standard and the new tentative appear in the 1958 Book of ASTM Standards, Part 5.

This report has been submitted to letter ballot of the committee, which consists of 27 members; 26 members returned their ballots, of whom 25 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

D. G. Miller,
Chairman.

Robert G. Scott,
Secretary.

Editorial Note

Subsequent to the Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specification for:

Standard Strength Ceramic Glazed or Unglazed Clay Sewer Pipe (C 261 – 57 T), and Extra Strength Clay Pipe (C 200 – 57 T).

These recommendations were accepted by the Standards Committee on November 6, 1959, and the revisions of the specifications appear in the 1959 Supplement to Book of ASTM Standards, Part 5.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

REPORT OF COMMITTEE C-7
ON
LIME*

Committee C-7 on Lime held two meetings during the year: on June 24, 1958 in Boston, Mass., during the Annual Meeting of the Society, and on February 3, 1959, in Pittsburgh, Pa., during ASTM Committee Week.

The committee consists of 50 members, of whom 24 are classified as producers, 7 as consumers, and 18 as general interest, with 1 honorary member.

An informal symposium on the Soundness of Lime was held during the Pittsburgh meeting.

The committee has reactivated Subcommittee VIII on Nomenclature, with B. M. O'Kelly as chairman.

Representation on Other Committees.—Representation on Subcommittee 29 on Microchemical Apparatus of Committee E-1 on Methods of Testing was dropped. P. L. Rogers was appointed to represent Committee C-7 on Subcommittees 10 and 11 of Committee E-1, while Robert K. Thomas represents the committee on Subcommittee 15 of Committee E-1.

V. S. Tadsen was appointed to represent Committee C-7 on Committee C-14 on Glass and Glass Products.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications as appended hereto:[1]

Tentative Specifications for:

Quicklime and Hydrated Lime for Hypochlorite Bleach Manufacture, and

Pozzolans for Use with Lime.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Specifications for Quicklime for Structural Purposes (**C 5 – 26**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

New Section.—Add a new Section 2 entitled "General Requirements" to read as follows, renumbering subsequent sections accordingly:

> 2. *General Requirements.*—(*a*) Quicklime shall be slaked and aged in accordance with the printed directions of the manufacturer. The resulting lime putty shall be stored until cool.
>
> (*b*) Lime putty prepared in accordance with Appendix A4 (*a*) and adjusted to standard consistency according to Sections 6(*b*) and (*c*) of Methods C 110 shall show no pops or pits when tested in accordance with the method prescribed in Section 14 of Methods C 110.
>
> (*c*) Lime putty prepared as above shall have a plasticity figure of not less than 200.

Appendix.—In Paragraph A3 insert a new first sentence to read: "Quicklime shall be slaked in accordance with the printed directions of the manufacturer.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

Where such directions are not provided, proceed as follows."

Revise Paragraph A4, Item (*a*) to read as follows:

(*a*) *White Coat.*—After slaking and aging finishing quicklime according to the printed directions of the manufacturer, the putty shall be stored until cool. If no printed directions are provided by the manufacturer, the putty shall be prepared for use as follows:

After the action has ceased, run off the putty through a No. 10 (2000-micron) sieve, and store for a minimum of two weeks.

AMERICAN STANDARDS

The committee recommends all specifications and methods of test under its jurisdiction for approval as American Standard by the American Standards Assn.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

SUBCOMMITTEE ACTIVITIES

Subcommittee VIII on Nomenclature (B. M. O'Kelly, chairman) is now working on definitions of limestone, dolomitic limestone, lime, and dolomitic lime.

Special Task Committee (J. E. Gray, chairman).—A Special Task Committee, consisting of J. E. Gray, chairman, Ralph B. Vogel and Francis J. Mardulier, was appointed to determine the need for specifications for limestone for use in agriculture and industry.

This report has been submitted to letter ballot of the committee, which consists of 51 members; 36 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. A. MURRAY,
Chairman.

L. E. JOHNSON,
Secretary.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-8
ON
REFRACTORIES*

Committee C-8 on Refractories held two meetings during the year: at Bedford Springs, Pa., on October 2, 1958, and at Pittsburgh, Pa., on February 6, 1959.

The committee consists of 45 members, of whom 24 are classified as producers, 16 as consumers, and 5 as general interest members.

The committee takes cognizance of the untimely death of Samuel Zerfoss, National Bureau of Standards, who was a faithful member of the committee.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods and specifications as appended hereto:[1]

Methods of Test for:

Size and Bulk Density of Insulating Fire Brick,
Reheat Change of Carbon Brick and Shapes,
Thermal Conductivity of Plastic Refractories, and
Resistance to Thermal Spalling of Silica Brick.

Specifications for:

Steel Pouring Pit Refractories, and
Insulating Fire Brick for Linings of Industrial Furnaces Operated with a Neutral or Oxidizing Atmosphere.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

Standard Method of Testing Refractory Brick Under Load at High Temperatures (C 16 – 49):[2]

Table I.—Revise to read as shown in the accompanying Table I.[3]

Standard Method of Test for Reheat Change of Refractory Brick (C 113 – 46):[2]

Table I.—Revise to read as shown in the accompanying Table II.[3]

REVISION OF TENTATIVE REVISION

The committee recommends revision as follows of the tentative revisions issued in June, 1958, of the Standard Definitions of Terms Relating to Refractories (C 71 – 55):[2]

Revise the following definitions to read as indicated:

Raw Refractory Dolomite.—Natural dolomite which is suitable for use as a refractory material.

Calcined Refractory Dolomite.—Raw refractory dolomite which has been heated to a temperature sufficiently high and for a long enough time to decompose the carbonate structure and remove volatile constituents.

Dead-Burned Refractory Dolomite.—Raw refractory dolomite which has been heated with or without additives to a temperature sufficiently high and for a long enough time to decompose the carbonate structure so as to form calcium oxide and periclase in a matrix that provides resistance to subsequent hydration and recombination with carbon dioxide.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 5.

[2] 1958 Book of ASTM Standards, Part 5.

[3] The revised table is not included in this report but appears in the revised Method of Test, see 1959 Supplement to Book of ASTM Standards, Part 5.

WITHDRAWAL OF TENTATIVE REVISION

The committee recommends the withdrawal of the tentative revisions issued June 1957 of Section 4 of the Standard Method for Basic Procedure in Panel Spalling Test for Refractory Brick (**C 38 – 58**).[2]

EDITORIAL REVISIONS

The committee recommends editorial changes as indicated in the following Standards:

Standard Method of Test for Bonding Strength of Air-Setting Refractory Mortar (Wet Type) (C 198 – 47):[2]

Title.—Revise to read: "Cold Bonding Strength of Air-Setting Refractory Mortar (Wet Type)."

Glossary of Terms Relating to Refractories:

The committee recommends that definitions for five terms be reinstated in the Glossary of Terms Relating to Refractories that appears in the Manual of ASTM Standards on Refractory Materials issued in 1957. This Glossary is a useful compilation made by The Refractories Institute and is not an ASTM standard but is published as useful information. The terms are: abutment, acid proof brick, American bond, arch, and arch brick. Definitions for these terms at present appear in the C-8 Manual as a tentative revision to Standard Definitions of Terms Relating to Refractories (C 71 – 55) issued June 1956 but were subsequently withdrawn from Definitions C 71 in June 1958.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (R. P. Stevens, chairman) has received an offer from W. O. Brandt to revise the present survey of the copper industry. J. L. Sullivan and R. B. Engdahl authored a paper that provides valuable data for inclusion in the survey on incinerators now published in the Committee C-8 Manual. Work is in progress on surveys of blast furnaces, electric furnaces, lead industry, and portland cement.

Subcommittee II on Research (R. B. Sosman, chairman) is undertaking to subdivide and revise several of the research problems described in "Some Unsolved Problems," ASTM BULLETIN, August, 1954.

Subcommittee III on Tests (S. M. Phelps, chairman) has approved for publication the new tentative methods of test reported earlier as well as the tentative revisions of test methods.

Section A on Load (J. D. Sullivan, chairman) has recommended revision of the heating table in Method C 16 to include two new schedules. This change permits a test method for some newer refractories, such as improved basic and neutral brick, that will stand higher temperatures than shown in the older schedules dating back to 1949.

Section B on Spalling (L. J. Trostel, chairman) recommended the withdrawal of the revision to Method C 38 involving a substitution of a mixture of straight kaolin and alumina because of conflicting results obtained in several laboratories. The problem of sticking of the mortar is being further studied. The section also recommended a new hot-plate method for measuring the spalling of silica brick.

Section C on Temperature (J. L. Carruthers, chairman) continued work on the design and development of a standard PCE furnace in the Refractories Section of the National Bureau of Standards. L. E. Mong presented a paper describing evaluation of four furnaces and the proposed PCE furnace.

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Section D on Precision and Tolerance (W. Duckworth, chairman) has started an investigation on the size tolerances in Specifications C 63, C 64, C 106, and C 153. The section will first ascertain size variation in appropriate brick as currently produced.

Section E on Chemical Analysis (H. A. Heiligman, chairman) has assisted in the preparation of two new standard samples of low- and high-alumina silica brick now available and certified by the National Bureau of Standards. The section also assisted in preparing a standard sample of crushed chrome ore. The certification of this sample has not yet been announced.

The Task Force on Hydration (G. R. Eusner, chairman) has agreed on a procedure believed generally acceptable for hydration resistance of basic brick, magnesite grain, and oiled dolomite. A procedure has been suggested for oiled dolomite grain. Interlaboratory data are being obtained to support this new procedure.

Section F on Refractory Insulation (W. L. Stafford, chairman) has proposed the method for bulk density and the specification for insulating fire brick for furnace linings submitted with this report. Currently, investigations are being conducted for use in classifying a Group 30 (3000 F) insulating fire brick.

Section G on Porosity and Permanent Volume Change (L. C. Hewitt, chairman) has assisted in the preparation of methods for size and bulk density of insulating fire brick, reheat change of carbon brick, and in revision of heating schedules of Methods C 16 and C 113. They also developed for information a method of determining the bulk specific gravity of granular hydratable refractory materials.

Section H on Mortars and Plastic and Castable Refractories (G. H. Anthony, chairman) originated the change in title of Method C 198 to indicate cold bonding strength. It has under consideration a test for bonding strength of mortars after firing and subsequently cooling the specimens.

Subcommittee IV on Heat Transfer (C. L. Norton, Jr., chairman) developed the method of thermal conductivity of plastic refractories submitted with this report. It is at present reviewing data relative to extending the scope of Method C 201 to include material having a thermal conductivity as high as silicon carbide.

Subcommittee VI on Nomenclature (A. W. Allen, chairman) recommended the revisions in Definitions C 71 referring to the three types of dolomite as herein reported. Under consideration are terms from the glossary appearing in the C-8 Manual. The subcommittee has proposed recommendations concerning a compilation of Terms Relating to Refractory Materials circulated by ISO/ Technical Committee 33 on Refractories.

Subcommittee VII on Specifications (J. D. Sullivan, chairman) approved two specifications submitted with this report. The section has been working with Section III D on the size tolerance in Specifications C 63, C 64, C 106, and C 153 and has recommended that these four designations be reapproved without change.

The Ad Hoc Group on Steel Pouring Pit Refractories has completed the specification on steel pouring pit refractories submitted with this report.

Subcommittee VIII on Editorial (W. R. Kerr, chairman) has reviewed all new methods and specifications submitted with this report.

Subcommittee IX on Classifications (J. A. Kayser, chairman) is preparing a new classification covering basic brick.

Progress has been made in reaching fundamental agreement about the kind of classification that should be written.

Subcommittee XI on Special Refractories (A. Maupin, chairman) is studying results of test data on pure grade sintered mullite. Interlaboratory tests were made at eight laboratories by several different methods. It is hoped that a classification of mullite refractories will result from this cooperative study of mineralogical content of the sample. The group has circulated a load test for mullite refractories to all manufacturers of mullite asking for comments. Progress is being made in the direction of a standard load test for mullite refractories.

Subcommittee XII on Carbon Refractories (E. B. Snyder, chairman) completed cooperative tests on permeability and is preparing a method for approval. The permeability work is being expanded to include fireclay, silica, and basic refractories as well as carbon. The group has started interlaboratory tests with the aim of developing a standard test for alkali attack on carbon refractories, and a task group under R. W. Limes is investigating methods of determining thermal conductivity of carbon refractories.

Subcommittee XV on Basic Granular Refractories (J. J. Hazel, chairman) is conducting tests on nine samples of burned dolomite representing material from Missouri, Illinois, Ohio, and West Virginia. Over ten separate properties are being investigated and the subcommittee hopes to develop a suitable test for refractoriness of burned dolomite from this work.

This report has been submitted to letter ballot of the committee which consists of 45 members; 45 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. J. HAZEL,
Chairman.

L. J. TROSTEL,
Secretary.

REPORT OF COMMITTEE C-9
ON
CONCRETE AND CONCRETE AGGREGATES*

Committee C-9 on Concrete and Concrete Aggregates held two meetings during the year: on June 25, 1958, during the Annual Meeting of the Society in Boston, Mass., and on December 5, 1958, at Purdue University, Lafayette, Ind.

At its June meeting, the committee adopted a memorial to Mr. H. S. Mattimore, a member of the committee since 1918 and an Honorary Member of the committee, who died on March 3, 1958. At its December meeting, the committee adopted memorials to Robert F. Blanks, a member of the committee since 1935, who died on July 14, 1958; and to Charles W. Allen, a member since 1946, who died on October 10, 1958.

At its December meeting, the Executive Subcommittee elected A. A. Levison and L. W. Teller to Honorary Membership.

The Sanford E. Thompson Award will be made this year to George Verbeck of the Research and Development Laboratories of the Portland Cement Assn. for his paper entitled "Carbonation of Hydrated Portland Cement," which was presented at the Second Pacific Area National Meeting of the Society, September 1956, and published in *ASTM STP No. 205*.

NEW TENTATIVES

The committee recommends for publication as tentative the following method and specification as appended hereto:[1]

Proposed Tentative Method of Test for:

Determining the Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction.

Proposed Tentative Specifications for:

Cotton Mats for Curing Concrete.

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 56 T):[2]

Section 2(a).—Add the following sieves: No. 5, $\frac{5}{16}$ in., $\frac{5}{8}$ in., $1\frac{1}{4}$ in.

Section 4.—Delete from Paragraph (*b*) the gradings shown as Alternate A and Alternate B, and the note following Alternate B.

To Paragraph (*c*) add the following: "When the $\frac{3}{8}$ to $\frac{3}{4}$ in., $\frac{3}{4}$ to $1\frac{1}{2}$ in., or $1\frac{1}{2}$ to $2\frac{1}{2}$ in. test sample specified in Paragraph (*b*) cannot be prepared due to absence of one of the two sizes of aggregate shown for each, the size available shall be used to prepare the sample tested."

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

Section 7.—Revise Paragraph (*b*) to read as follows:

> (*b*) After the sodium sulfate or magnesium sulfate solution has been removed, each fraction of the sample shall be dried to constant weight at 221 to 230 F (105 to 110 C), weighed, and, except in the case of ledge rock, the fine aggregate shall be sieved over the same sieve on which it was retained before the test, and the coarse aggregate shall be sieved over the sieve shown below for the appropriate size of particle. The particles retained on each sieve shall be weighed and the weight recorded.

Size of Aggregate	Sieve Used to Determine Loss
2½ to 1½ in.	1¼ in.
1½ to ¾ in.	⅝ in.
¾ to ⅜ in.	5/16 in.
⅜ in. to No. 4	No. 5

Delete Paragraph (*c*), relettering the following paragraph as (*c*).

Section 9(*e*).—Change the reference to Section 7(*d*) to read "Section 7(*c*)."

Tentative Specifications for Waterproof Paper for Curing Concrete (C 171 – 58 T):[2] Revise as appended hereto.[3]

Tentative Methods of Sampling and Testing Fly Ash for Use as an Admixture in Portland Cement Concrete (C 311 – 57 T):[2]

Section 27.—In Paragraph (*b*), lines 6 and 7, change "replacing an equivalent weight of the sand" to read "replacing an equivalent weight (oven-dry basis) of the sand."

In Paragraph (*c*), in line 5 change "twice" to "2.0 times." Add to the end of the last sentence the following: "except that for the freezing and thawing test the mixture specified in Paragraph (*b*) shall be compared with that specified in Paragraph (*a*)."

Tentative Specifications for Lightweight Aggregates for Structural Concrete (C 330 – 53 T):[2]

Section 5(*c*).—Change "*Unburned or Underburned*" to "*Clay*" in heading and in sentence, and delete "in expanded clay, shale, or slate aggregates."

Section 6(*a*).—Revise to read as follows:

> (*a*) *Compressive Strength and Unit Weight.*—It shall be possible to produce structural concrete, using the lightweight aggregates under test, such that one or more of the compressive strength requirements in the following table will be satisfied without exceeding the corresponding maximum unit weight values. Intermediate values for strength, and corresponding unit weight values, may be established by interpolation.

Average 28-Day Compressive Strength, min, psi	Average Unit Weight, max, lb per cu ft
4000	115
3000	110
2000	105

Section 7(*f*).—In the heading change "*Unburned or Underburned*" to read "*Clay.*"

Section 8.—Delete Paragraph (*c*), relettering subsequent paragraphs accordingly. Change references to renumbered paragraphs as necessary in Section 6(*b*) and (*c*) and in Section 8(*d*).

Tentative Specifications for Lightweight Aggregates for Concrete Masonry Units (C 331 – 53 T):[2]

Section 5(*c*).—In the heading and in sentence change "*Unburned or Underburned*" to read "*Clay,*" and delete "in expanded clay, shale, or slate aggregates."

Section 7(*f*).—In the heading change "*Unburned or Underburned*" to read "*Clay.*"

REVERSION OF STANDARD TO TENTATIVE

The committee recommends reversion of the Standard Method of Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified Cube Method) (**C 116 – 49**)[2] to tentative pending revision.

---

[3] The revised tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 4.

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 - 56 T):[2]

Section 2.—In line 5, change "Section 15" to read "Sections 15 and 17."

Tentative Method of Test for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading) (C 293 - 57 T):[2]

Section 2.—In lines 3 and 4 change "Section 15" to read "Sections 15 and 17."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 - 57):[2]

Section 1.—Add a new Note 2 to read as follows, renumbering subsequent notes accordingly:

NOTE 2.—The methods of molding specimens (Sections 5 and 10) are not considered applicable if the characteristics of the concrete are such that rodding will not produce specimens of density comparable to that of the concrete in the structure. Methods for the consolidation of such specimens are under development.

Standard Specifications for Concrete Aggregates (C 33 - 57):[2]

Section 4(a).—Delete the clause "each determined on independent samples complying with the grading requirements of Section 3."

Table II.—Add size No. 8 from Specifications D 448 and the sieve analysis limits therefor. This will involve adding an extra column to the table for the No. 16 (1190-micron) size.

Section 8(a).—Delete the clause "determined on a sample complying with the designated grading requirements of Section 7."

Section 12.—Add a second sentence to the introductory paragraph to read as follows, changing the colon at the end of the present introductory sentence to a period:

The required tests shall be made on test samples which comply with requirements of the designated test methods and are representative of the grading which will be used in the concrete. The same test sample may be used for sieve analysis and for determination of material finer than the No. 200 sieve. Separated sizes from the sieve analysis may be used in preparation of samples for soundness or abrasion tests. For determination of all other tests and for evaluation of potential alkali reactivity where required, independent test samples shall be used.

Standard Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 - 57):[2]

Section 4.—Replace Paragraph (*a*) with the following new Paragraphs (*a*) and (*b*), relettering the present Paragraph (*b*) as (*c*):

4. (*a*) *Core Specimens, Pavement Thickness.*—A core specimen for the determination of pavement thickness shall have a diameter of at least 4 in.

(*b*) *Core Specimens, Compressive Strength.*—A core specimen for the determination of compressive strength should have a diameter at least three times the maximum nominal size of the coarse aggregate used in the concrete, and in no case shall the diameter of the specimen be less than twice the maximum nominal size of the coarse aggregate. The length of the specimen, when capped, shall be as nearly as practicable twice its diameter.

Standard Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78 – 57):[2]

Section 2.—In the second line change "Section 15" to read "Sections 15 and 17."

Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregates (C 127 – 42):[2]

Section 1(a).—Delete the words "at room temperature."

Section 2(b).—Change "No. 4 mesh" to read "No. 6 or No. 8 mesh."

Section 3.—In the first sentence change "$\frac{3}{8}$-in. sieve" to read "No. 4 sieve." Delete the last sentence.

Section 4.—In Paragraph (*a*) delete the words "at 59 to 77 F (15 to 25 C)."

In Paragraph (*b*), at the end of the sentence add "at 68 to 77 F (20 to 25 C)."

Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 57):[2]

Section 1(a).—Delete the words "at room temperature."

Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 57):[2]

Section 1.—Add a new Note 2 to read as follows, renumbering subsequent notes accordingly:

NOTE 2.—The methods of molding specimens (Sections 9 and 14) are not considered applicable if the characteristics of the concrete are such that rodding will not produce specimens of density comparable to that of the concrete in the structure. Methods for the consolidation of such specimens are under development.

Section 14(a).—Change the second sentence to read "The concrete shall be placed in layers approximately 3 in. deep, each layer consolidated by rodding (Note 10)."

New Note.—Add a new Note 10 to read as follows:

NOTE 10.—When the depth of the specimen is 4 in. or less, it may be placed in one layer or two equal layers at the option of the operator.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

Unit Weight of Aggregate (C 29 – 55 T),
Organic Impurities in Sands for Concrete (C 40 – 56 T),
Lightweight Pieces in Aggregate (C 123 – 57 T),
Clay Lumps in Natural Aggregates (C 142 – 55 T),
Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete (C 156 – 55 T),
Volume Change of Cement Mortar and Concrete (C 157 – 54 T),
Air Content of Freshly Mixed Concrete by the Pressure Method (C 231 – 56 T),
Comparing Concretes on the Basis of the Bond Developed with Reinforcing Steel (C 234 – 57 T),
Scratch Hardness of Coarse Aggregate Particles (C 235 – 57 T),
Potential Reactivity of Aggregates (Chemical Method) (C 289 – 57 T),
Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 – 57 T),
Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 – 57 T),
Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292 – 57 T),
Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 – 57 T),
Potential Volume Change of Cement-Aggregate Combinations (C 342 – 55 T),
Ball Penetration in Fresh Portland Cement Concrete (C 360 – 55 T), and
Rate of Hardening of Mortars Sieved from Concrete Mixtures by Proctor Penetration Resistance Needles (C 403 – 57 T).

Tentative Specifications for:

Lightweight Aggregates for Insulating Concrete (C 332 - 56 T), and

Fly Ash for Use as an Admixture in Portland Cement Concrete (C 350 - 57 T).

EDITORIAL REVISIONS

The committee recommends editorial changes as indicated in the following tentatives and standard:

Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 - 56 T):[2]

Section 2(e).—In line 9, delete the plus-minus sign after "3 F."

Tentative Method of Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231 - 56 T):[2]

Section 5(b).—In the next to the last line delete the word "fine."

Standard Method of Test for Slump of Portland Cement Concrete (C 143 - 58):[2]

Section 2(a).—Add the following at the end of the paragraph: "The mold may be constructed either with or without a seam. When a seam is required, it should be essentially as shown in Fig. 1."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Group I, Administration (I. L. Tyler, chairman).—Subcommittee I-b on Papers and Symposia has arranged two sessions of technical papers, four papers for each session, for the Annual Meeting at Atlantic City in June.

Subcommittee I-c has completed its recommendations for the Sanford E. Thompson Award as noted earlier in the report.

Subcommittee I-d, Editorial and Definitions, has reviewed and corrected C-9 standards and definitions for publication.

Group II on Research (L. E. Gregg, chairman).—The scope of Subcommittee II-a was revised to broaden its activities and particularly to give assistance to other subcommittees in the application of statistical methods in planning research programs and the statistical evaluation of data.

Subcommittee II-b prepared the Proposed Tentative Method of Test for Determining the Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction referred to earlier in this report.

The subcommittee arranged for the sampling of hardened concrete from a number of sections of the McPherson Test Road in Kansas, which has been in service ten years and offers unusual opportunities for realistic study of deterioration caused by reactive aggregates. These specimens were obtained through the cooperation of the State Highway Commission of Kansas. Sections from some of the specimens have been sent to several cooperating laboratories for chemical and microscopical analyses. It is hoped that these studies will explain the factors that cause unusual expansion and cracking in concrete made with certain sand-gravel aggregates.

Subcommittee II-c is developing two new test methods for the measurement of the static modulus of elasticity of concrete in compression and the measurements of creep of concrete in compression.

Extension of the information given in Descriptive Nomenclature C 294 and Recommended Practice C 295 to meet the needs of laboratory personnel unversed in petrographic methods was discussed by Subcommittee II-f. A revision of these standards was considered de-

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

sirable in some respects, and the preparation of a third document, which would explain the material presented in the other two, is planned.

Subcommittee II-g is drafting a recommended practice for microscopical determination of air void content and the specific surface and spacing factor of the air void system on hardened concrete.

The new Subcommittee II-h on Concrete for Radiation Shielding, established by action of the Executive Subcommittee at the December meeting, initiated a thorough study of available data to secure necessary information for the development of performance requirements for high-density aggregates and for concretes made with such aggregates in order to determine needed additional research in this field. Specific problems are being assigned to members of the subcommittee for determination of properties of high-density aggregates, particular properties of fresh and of hardened concretes containing these aggregates, and general aspects of problems of radiation shielding.

An Ad Hoc Committee was established to make an intensive study and evaluation of epoxy resins and similar materials serving as adhesives or bonding agents, as they relate to the scope of Committee C-9, to determine whether ASTM has a legitimate interest in this field which might lead to specifications, methods of test, or recommended practices.

Group III on Specifications and Test Methods (Stanton Walker, chairman).—The results of the principal subcommittee activities are adequately covered in that portion of this report dealing with action on standards. Some special projects deserve mention:

1. Subcommittee III-a is developing procedures for molding specimens from low slump concrete.
2. Subcommittee III-c has prepared for publication as information a tentative method of test for cement content of freshly mixed concrete.
3. Subcommittee III-e has undertaken to expedite agreement on standards within its scope and under joint jurisdiction of two or more committees by the organization of joint committees.
4. Subcommittee III-f is interested in taking a more direct part in adapting test methods to lightweight aggregates and concrete.
5. Subcommittee III-i has under consideration revisions in Specifications C 94, including the development of criteria for evaluating the efficiency of concrete mixers.

Some realignment of subcommittee activities is in process. The test methods of Subcommittee III-k have been assigned to Subcommittee III-l, and those of Subcommittee III-j to Subcommittee III-c.

This report has been submitted to letter ballot of the committee, which consists of 178 active members commanding 130 votes plus 8 honorary members having voting privileges; 166 members returned their ballots, of whom 165 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. H. PRICE,
Chairman.

BRYANT MATHER,
Secretary.

REPORT OF COMMITTEE C-11
ON
GYPSUM*

Committee C-11 on Gypsum held two meetings during the year: in Boston, Mass., on June 26 and 27, 1958, and in Pittsburgh, Pa., on February 2 and 3, 1959.

The committee consists of 36 members, of whom 34 are voting members; 13 are classified as producers, 10 as consumers, 11 as general interest, and 2 as consulting members.

NEW TENTATIVE

The committee recommends for publication as tentative the Specifications for Gypsum Backing Board as appended hereto.[1]

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision[2] comprising new Sections 42 to 45 of the Standard Methods of Testing Gypsum and Gypsum Products (**C 26 - 56**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Definitions of Terms Relating to Gypsum (C 11 - 58):[2]

Gypsum Wallboard.—Change the Note to read as follows:

NOTE: *Type X, Gypsum Wallboard.*—"Type X" (Special Fire Retardant) designates gypsum wallboard, complying with ASTM Specifications C 36, that provides at least: (1) 1-hr fire retardant ratings for ⅝ in. thick or (2) ¾-hr fire-retardant ratings for ½ in. thick gypsum wallboard applied in single layer nailed application on each face of load-bearing wood framing members when tested in accordance with the requirements of Methods of Fire Tests of Building Construction and Materials (ASTM Designation: E 119). Consult manufacturers for independent test data on assembly particulars, materials, and ratings for other types of construction.

Specifications for Gypsum Plasters (C 28 - 58):[2]

Section 9.—Change "32 hr" to read "15 hr."

Section 13.—Change "16 hr" to read "9 hr."

Section 17.—In the first sentence, change "10 hr" to read "9 hr."

Section 23.—Change to read as follows:

23. *Sampling.*—At least 1 per cent of the packages, but not less than 5 packages, shall be sampled. Packages to be sampled shall be selected at random. Samples shall be taken both from the outer portion and the center of each package. The materials so obtained shall be thoroughly mixed to provide a composite sample of not less than 15 lb. This composite sample shall be placed immediately in a clean, dry, airtight container for delivery to the laboratory.

Section 24.—Delete this section.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specifications for Inorganic Aggregates for Use in Gypsum Plaster (C 35 – 57 T)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 34 members; 19 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. W. JOSEPHSON,
Chairman.

R. H. FABER,
Secretary.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-12
ON
MORTARS FOR UNIT MASONRY*

Committee C-12 on Mortars for Unit Masonry held two meetings during the past year: on February 5, 1959 in Pittsburgh, Pa., and June 26, 1958 in Boston, Mass.

At the present time the committee consists of 52 members, of whom 23 are classified as producers, 9 as consumers, and 19 as general interest members, with 1 consulting member.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specifications for Mortar for Unit Masonry (**C 270 – 57 T**)[1] and continuation of the specification as tentative.

Section 2 (a).—Add a new paragraph to read as follows: "*Slag Cement*.—Type S or SA of the Specifications for Slag Cement (ASTM Designation: C 358)."

Table II.—Revise the heading of column 2 to read, "Parts by Volume of Portland Cement or Portland Blast-Furnace Slag Cement."

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Specifications for Mortar for Reinforced Brick Masonry (**C 161 – 44 T**).[1]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Aggregate for Masonry Mortar (**C 144 – 52 T**),[1] and

Aggregates for Masonry Grout (**C 404 – 57 T**).[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Research and Methods of Test (P. L. Rogers, chairman) completed work on a procedure for quantitative evaluation of efflorescence which was published in the ASTM BULLETIN.[3] Current studies are being made on the effect of waiting time on water retention of mortars.

Subcommittee III on Specifications (H. C. Plummer, chairman) is preparing a proposed tentative specification for mortar for reinforced masonry which will replace Specification C 161 – 44 T now being withdrawn.

Subcommittee IV on Aggregates (D. L. Bloem, chairman) is conducting a cooperative series of tests to evaluate the influence of sand gradation upon the properties of masonry mortar.

This report has been submitted to letter ballot of the committee, which consists of 48 members; 33 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. COPELAND,
Chairman.

C. U. PIERSON, JR.,
Secretary.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] 1958 Book of ASTM Standards, Part 4.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[3] P. L. Rogers, "A Method of Test for Potential Efflorescence of Masonry Mortar," ASTM BULLETIN, No. 235, Jan., 1959, p. 31 (TP 1).

REPORT OF COMMITTEE C-13

ON

CONCRETE PIPE*

Committee C-13 on Concrete Pipe held one meeting during the year, in Chicago, Ill., on November 21, 1958.

The committee consists of 40 members, of whom 19 are classified as producers, 13 as consumers, and 8 as general interest members.

We report with regret the death of W. W. Horner of Horner & Shifrin, St. Louis, Mo. Mr. Horner, a Past-Chairman of the committee, was an active member for many years and had contributed greatly to the work and success of the committee.

The Advisory Committee appointed a separate subcommittee to act on its recommendations concerning the separation of the method of tests from the body of the specifications.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 2 new tentatives and is recommending the revision of 2 tentatives and 2 standards, the withdrawal of 1 tentative, and the continuation of 1 tentative without revision. The standards and tentatives affected, together with the revisions recommended, are covered in detail in the Appendix to this report.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Non-Reinforced Sewer Pipe (C. A. Bluedorn, chairman) recommended the revisions of Specifications C 14 noted in the Appendix to this report.

The following matters will be under consideration during the coming year: (1) curing; (2) deletion of the sand bearing test; (3) review of specification C 14 with regard to standardization of similar items between specifications, and editorial and organization improvements; (4) possible inclusion of the bell or groove end of pipe in the permeability test; and (5) study of the wall thicknesses of pipe to determine if the wall thicknesses now shown are realistic or whether they should be revised. This will probably entail an actual research project.

Subcommittee II on Reinforced Sewer and Culvert Pipe (E. F. Bespalow, chairman) recommended the revisions of Specifications C 76 referred to in the Appendix to this report.

Under consideration during the coming year will be (1) increasing or enlarging the scope of the tables to include other sizes, changes in wall thickness and other factors that might be brought to the attention of the subcommittee during the year, and (2) deletion of the sand bearing test.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] See p. 398.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on Irrigation and Drain Pipe (A. F. Pillsbury, chairman) recommended the numerous revisions to Specifications C 118. Consideration of specifications for monolithic concrete conduit will be continued.

Subcommittee IV on Low Head Pressure Pipe (H. G. Curtis, chairman) recommended the revisions to Specifications C 361 covered in the Appendix to this report.

Subcommittee V on Low-Head Internal Pressure Sewer Pipe (M. Butler, chairman) has recommended that Specifications C 362 be withdrawn and that Subcommittee V be dissolved.

Subcommittee VI on Drain Tile (P. W. Manson, chairman) reported that the new Tentative Specifications C 412 have been favorably received but that they have not been in use a sufficient length of time to warrant recommendations for change.

Subcommittee VII on Perforated Pipe (H. Allen, chairman) submitted the proposed Tentative Specification for Perforated Concrete Pipe appended to this report.

Special Subcommittee on Rubber Gaskets (C. A. Bluedorn, chairman) submitted the proposed Tentative Specification for Flexible Watertight Rubber Type Gasket Joints for Circular Concrete Sewer and Culvert Pipe appended to this report.

Special Subcommittee on Absorption (J. W. Johnson, chairman) has recommended that the subcommittee be dissolved.

Special Subcommittee on Method of Tests (H. Allen, chairman).—This subcommittee has been appointed to implement the following recommendations made by the Advisory Committee in regard to methods of tests:

1. That the method of tests be separated from the body of the specifications while recognizing that the tests as separated should be printed as a separate section after the body of the specifications.
2. There should be a consolidation of all the tests in this separate test section.
3. Several of the tests are now written differently in the different specifications under the jurisdiction of Committee C-13. These should be reviewed and rewritten into one clear, simple, concise statement.

This report has been submitted to letter ballot of the committee, which consists of 40 voting members; 37 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

HOWARD F. PECKWORTH,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR CONCRETE PIPE

This Appendix contains the recommendations affecting certain standards covering concrete pipe which are referred to earlier in this report.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications as appended hereto:[1]

Tentative Specifications for:

Flexible Watertight Rubber Type Gasket Joints for Circular Concrete Sewer and Culvert Pipe, and
Perforated Concrete Pipe.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Reinforced Concrete Culvert, Storm Drain, and Sewer Pipe (C 76 – 57 T):[2]

Section 10.—Delete the first six words of the second sentence.

Section 25.—In Paragraph (*a*) revise the second sentence to read as follows: "The internal diameter of 27- to 108-in. pipe shall not vary more than ±1 per cent or $\frac{3}{8}$ in., whichever is greater, from the nominal diameter."

Delete Paragraph (*c*) and replace with the following paragraph:

(*c*) Where single cage circular reinforcement is used in either circular pipe or elliptical pipe and for all elliptical cage reinforcement, a steel area that is not less than 97 per cent of that area shown in Tables I to V will be considered as meeting the required steel area. Where two circular cage reinforcements (one inner and one outer cage) are used, again referring to Tables I to V, the inner cage steel area may vary to the lower limit of being not less than 85 per cent of the same design elliptical steel area; and the outer cage steel area may vary to the lower limit of being not less than 64 per cent of the same design elliptical steel area; provided that in no case the total steel area of the inner cage plus the outer cage be less than 153 per cent of the same design elliptical steel area.

Tentative Specifications for Reinforced Concrete Low-Head Pressure Pipe (C 361 – 57 T):[2]

Section 9.—In Paragraph (*a*) the reference to Paragraph (*b*) on line 14 should be changed to Paragraph (*c*).

In Paragraph (*b*) delete the third sentence and add the following material to the second sentence:

. . . or for concrete of a consistency too stiff for compaction by rodding or internal vibration, the following alternate method may be used for cylinder fabrication: A mold as described in Method C 31 shall be bolted to the top of a vibrating metal table. Concrete shall be placed in 3-in. lifts within the cylinder. Each lift shall be externally vibrated on the table by a high frequency type form vibrator rigidly attached to the underside of the metal table top. The vibrator shall operate at a speed of at least 8000 rpm during vibration. A 10-lb cylindrical hammer $\frac{1}{4}$ in. less in diameter than the inside of the mold shall be placed on the surface of each concrete lift during vibration. Vibration shall continue until cement paste begins to ooze up around the bottom edge of the hammer. This procedure shall be repeated until the form is filled. Where 3 by 6-in. cylinders are allowed for

---

[1] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 5.

[2] 1958 Book of ASTM Standards, Part 5.

tests, the method of vibration shall be as above, except the mold shall be filled in 2-in. lifts and the weight of the compacting hammer shall be 2.5 lb.

Add a new Paragraph (*c*) to read as follows:

(*c*) The average compressive strength of all cylinders tested at 28 days shall be not less than 4500 psi and not more than 10 per cent of the cylinders tested shall fall below 4500 psi. In no case shall any cylinder tested fall below 3600 psi. Where Type V cement is specified by the purchaser, the average compressive strength of all concrete cylinders tested at 28 days shall be not less than 3500 psi, and not more than 10 per cent of the cylinders tested shall fall below 3500 psi. In no case shall any cylinder tested fall below, 2800 psi.

These compressive strength requirements refer to standard 6 by 12-in. concrete test cylinders. Where the strength of 6 by 12-in. concrete test cylinders exceeds the capacity of the normal field testing machine (200,000 lb), 3 by 6-in. test cylinders will be permitted with correction for size of cylinder.

Section 13.—In Paragraph (*a*), in the table of physical requirements change the water absorption requirement from 7 days to 48 hr.

In Paragraph (*b*), Item (*7*), in line 26 change the reference to 7 days to 48 hr. Delete the last sentence and substitute the following: "The water absorption shall be taken as the increase in weight of the specimen divided by its original dry weight."

Section 17.—Delete the first sentence and substitute the following sentence: "If the splices are not welded, the reinforcement shall be lapped not less than 20 diameters for deformed bars and 40 diameters for plain bars and cold drawn wire."

Section 21.—In the third sentence, delete the words "6 by 12-in."

Section 23(a).—In the third sentence, delete the words "6 by 12-in."

REVISION OF TENTATIVE REVISION OF STANDARD

The committee recommends that the tentative revision of the Standard Specifications for Concrete Pipe for Irrigation or Drainage (C 118 – 56)[2] issued June, 1954 and modified June, 1958 be revised to read as follows:

Section 5.—Add a footnote as follows: "When alkali or sulfates are present, sulfate-resistant cement may be required when specified in advance of purchase."

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

TABLE I.—PHYSICAL TEST REQUIREMENTS.

Internal Diameter, in.	Minimum Shell Thickness, in.	Minimum Internal Hydrostatic Pressure, psi	Minimum Three-Edge-Bearing Load, lb per lineal ft
4	3/4	50	1200
5	3/4	50	1250
6	3/4	50	1300
8	7/8	50	1350
10	1	50	1400
12	1 1/8	45	1500
14	1 1/4	45	1600
15	1 3/8	45	1650
16	1 3/8	45	1700
18	1 1/2	45	1800
20	1 7/8	40	1850
21	2	40	1900
24	2 1/8	40	2000

Standard Specifications for Concrete Sewer Pipe (C 14 – 58):[2]

Section 29.—Delete and substitute the following:

29. The following information shall be clearly marked on each section of pipe:

(*a*) The pipe class,

(*b*) The date of manufacture,

(*c*) The name or trademark of the manufacturer, and

(*d*) Identification of the plant. Markings shall be indented on the pipe section or painted thereon with waterproof paint.

Standard Specifications for Concrete Pipe for Irrigation or Drainage (C 118 – 56):[2]

Heading for Sections 8, 9, 10, and 11.—Change from "Design and Permissible Variations in Dimensions" to "Design."

Section 8.—Delete and replace with the following: "8. *Requirements.*—Minimum design shall be in accordance with Table I."

Table I.—Delete and replace with a new table as shown in the accompanying Table I.

Section 9.—Revise to read as follows:

> 9. The internal diameter shall not be less than the nominal internal diameter by more than $\frac{3}{16}$ in. for pipe 4, 5 or 6 in. in diameter, by more than $\frac{1}{4}$ in. for pipe from 8 to 18 in. in diameter, and by not more than $\frac{5}{16}$ in. for pipe over 18 in. in diameter.

Section 10.—Delete this section, renumbering subsequent sections accordingly.

Section 11.—In the first sentence change the word "uniform" to "watertight." Delete the second sentence.

Heading for Sections 12, 13, 14 and 15.—Change from "Physical Properties and Tests" to "Physical Test Requirements."

Section 12.—Revise the first sentence of Paragraph (*b*) to read as follows: "(*b*) The specimens to be tested shall be selected at random by the purchaser or his representative at the place of manufacture, and shall be tested in advance of shipment."

In Paragraph (*c*) change the words "strength tests" to read "three-edge-bearing load test."

Revise Paragraph (d) to read as follows:

> (*d*) Pipe shall be acceptable under the load and hydrostatic test requirements when all test specimens conform to the test requirements. Should any of the test specimens fail to meet the test requirements, then the manufacturer will be allowed a retest on two additional specimens for each specimen that failed, and the pipe shall be acceptable only when all of the retest specimens meet the test requirements.

Delete Paragraph (*e*).

Section 13.—Renumber as Section 12 and change to read as follows:

> 12. *Three-Edge-Bearing Test.*—(*a*) Pipe shall be tested in a device as shown in Fig. 1, and individual pipe shall meet the minimum load requirements specified in Table I. The load shall be applied parallel to and along a vertical plane passing through the axis of the pipe. The lower bearings shall consist of two wooden strips with vertical sides, having their interior top corners rounded to a radius of approximately $\frac{1}{2}$ in. The strips shall be straight and shall be securely fastened to a rigid block at least 6 by 6 in. in cross-section. The interior vertical sides of the strips shall be parallel and spaced a distance apart of not more than 1 in. per ft. of pipe diameter, but in no case less than 1 in. If requested by the manufacturer or purchaser previous to the test, before the pipe is placed, a fillet of plaster of Paris, sand or other equally suitable material thick enough to compensate for the inequalities of the pipe barrel, but not exceeding 1 in. in thickness, shall be cast on the surface of the lower bearings. The pipe shall be placed upon the fillet while the plaster of Paris is still somewhat plastic. The upper bearing shall be a rigid wooden block, free from knots, and straight and true from end to end. The load shall be applied to this block through a metal beam of such dimensions that it will transmit the full load without appreciable deflection. A fillet of plaster of Paris, sand or other equally suitable material, not exceeding 1 in. in thickness, may also be cast along the length of the crown of the pipe to equalize the bearing. The upper bearing shall be brought ln contact while the plaster of Paris is still somewhat plastic. The upper and lower bearings shall extend the full length of the pipe exclusive of any bell (see Fig. 1).
>
> (*b*) If mutually agreed by the manufacturer or other seller and the purchaser, proven types of bearings such as hard rubber blocks or sand-filled high pressure hose may be used in lieu of wooden bearings as specified above.
>
> (*c*) In making the test, any mechanical or hand power device may be used in which the head that applies the load moves at such speed as to increase the load at a rate of 500 to 2000 lb per linear ft of pipe per min. The testing machine shall be substantial and rigid throughout so as to provide a uniform deflection throughout the full length of the pipe. The load shall be applied continuously until the strength specified in Table I is reached. The load per linear foot of pipe shall be calculated by dividing the total

recorded load by the laying length. The pipe shall not be allowed to stand under load longer than is required to apply the load and to observe and record it. The pipe shall be surface-dry when tested. Test shall not be made on frozen pipe or at freezing temperatures.

Section 14.—Renumber as Section 13 and change to read as follows:

13. *Hydrostatic Test.*—(*a*) The equipment for making the test shall be such that the section of pipe under test can be filled with water to the exclusion of air and subjected to the required hydrostatic pressure without there being enough leakage from the ends of the pipe to interfere with the test, and such that no appreciable circumferential compression is placed on the outside of the pipe shell.

(*b*) The test shall not be made when the temperature of the pipe, the air around the pipe, or the water within the pipe is below 33 F.

(*c*) A standardized pressure gage shall be connected close to the specimen and the water pressure shall be brought up to 10 psi in about 1 min, and held at this pressure for 10 min. In order to pass this portion of the test, there shall be no excessive leakage during this period through the shell of the pipe. Moisture appearing on the surface of the pipe in the form of patches, or beads adhering to the surface will not be considered as excessive leakage. Slow forming beads of water from pinholes that result in minor dripping, and slight pinhole spurts which will not interfere with the use of the pipe in service, will not be cause for rejection.

(*d*) After the water pressure has been held at 10 psi for 10 min, it shall be increased at a uniform rate to the pressure specified in Table I. No failure shall occur at this pressure.

Section 15.—Delete this section, renumbering subsequent sections accordingly.

Figure 1.—Add the following footnote: "NOTE.—This figure illustrates the method of applying load to the pipe and not the types of joints allowable."

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Specification for Reinforced Concrete Low Head Internal Pressure Sewer Pipe (**C 362 – 55 T**).[2] Upon the adoption of the new Tentative Specification for Flexible Watertight Rubber Type Gasket Joints for Circular Concrete Sewer and Culvert Pipe, Specifications C 362 will become obsolete.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specification for Concrete Drain Tile (**C 412 – 58 T**).[2]

REPORT OF COMMITTEE C-15
ON
MANUFACTURED MASONRY UNITS*

Committee C-15 held two meetings during the year: on June 27, 1958, in Boston, Mass., in conjunction with the Annual Meeting of the Society; and on February 3, 1959, in Pittsburgh, Pa., during ASTM Committee Week.

The committee consists of 68 members, of whom 34 are classified as producers, 17 as consumers, and 17 as general interest members.

Honorary Memberships.—During the past year, two members who have served long and faithfully on Committee C-15 and whose interest in the work of the committee continues to be most active despite the fact that circumstances prevent their regular attendance and participation in committee work, were elected to Honorary Memberships on Committee C-15. These members are: Theodore Irving Coe and Dalton G. Miller.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, the committee presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Specifications for:

Facing Brick (Solid Masonry Units Made from Clay or Shale) (C 216 – 57).

Revision of Tentative Specifications for:

Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126 – 55 T).

New Tentative Methods of Test for:

Drying Shrinkage of Concrete Block (C 426 – 58 T), and

Moisture Condition of Hardened Concrete by the Relative Humidity Method (C 427 – 58 T).

These recommendations were accepted by the Standards Committee on September 9, 1958. The tentative revisions, revised tentative, and new tentatives appear in the 1958 Book of ASTM Standards, Part 5.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the Standard Specifications for Clay Drain Tile (C 4 – 55)[1] be revised as appended hereto and reverted to tentative status.[2]

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specifications for Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126 – 58 T),[2] and continuation of the specifications as tentative.

Table III.—Under Grade *S* Units, change to the following tolerances for the dimensions shown for the "If Smaller" and the "One Lot" headings:

Specified Face Dimension	*If Smaller*	*Maximum Difference in One Lot*
5 1/16	3/32	3/32
7 3/4	4/32	5/32
11 3/4	5/32	6/32

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] 1958 Book of ASTM Standards, Part 5.

[2] The revised specifications appear in the 1959 Supplement to Book of ASTM Standards, Part 5.

Table V.—Change to the following permissible distortions for the dimensions shown:

Face Dimensions	*Grade*	*Maximum Permissible Distortion, in.*
5 1/16 by 11¾	*S*	2/32
3¾ by 11¾	*S*	2/32

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revision of Section 5 of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

Standard Specifications for:

Hollow Load-Bearing Concrete Masonry Units (C 90 – 52),[1]

Hollow Non-Load-Bearing Concrete Masonry Units (C 129 – 52),[1] and

Solid Load-Bearing Concrete Masonry Units (C 145 – 52).[1]

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Executive (J. W. Whittemore, chairman).—Problems concerning committee membership balance occupied the major portion of the subcommittee's work during the past year. It has been necessary not only to scrutinize all membership applications from producers more closely, but also to maintain a closer check on the interest and activity of present members, in order that the committee not have too high a percentage of inactive and only mildly interested members.

In view of the large number of standards under the jurisdiction of Committee C-15, the Executive Subcommittee reviewed more closely the functions of the Editorial Subcommittee and recommended its more active participation in reviewing all proposed tentative revisions, revisions of tentatives, and proposed new tentatives prior to their submittal to letter ballot. Thus, the task of maintaining uniformity of expression and presentation and conformity with ASTM format and style will become easier.

Subcommittee II on Clay Brick and Structural Clay Tile (J. A. Lee, chairman).—The subcommittee prepared the tentative revisions of the Standard Specifications for Facing Brick (C 216 – 57), which were accepted by the Administrative Committee on Standards during the year.

Two task groups were appointed to study certain special problems. One group is reviewing the Explanatory Notes following Specifications C 62 and C 216 with the intention of preparing recommendations which will bring them into agreement. Another task group is reviewing the Standard Specifications for Paving Brick (C 7 – 42) and is making a survey of production and marketing of paving brick to obtain data on which recommendations relative to revisions of Specifications C 7 could be based.

The subcommittee is continuing its study of the "Weathering Index" as a more precise measure of the effect of weathering on brick.

Subcommittee III on Concrete and Sand Lime Units (R. E. Copeland, chairman) completed the preparation of the Tentative Method of Test for Drying Shrinkage of Concrete Block (C 426 – 58 T) and the Tentative Method of Test for Moisture Condition of Hardened Concrete by the Relative Humidity Method (C 427 – 58 T).

The subcommittee has sponsored a series of cooperative tests at ten laboratories to investigate the agreement obtained using the new Tentative Method of Test for Drying Shrinkage of Concrete Block (C 426 – 58 T).

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

It is also planning to collect additional test data and data from use experience in order to obtain information that will enable it to recommend limits on the moisture content of concrete masonry units.

Subcommittee V on Water Retardant Materials for Masonry Units (C. C. Connor, chairman). has continued exploratory work which, it hopes, will lead to the development of standard methods of test for the effectiveness of water retardant materials. One task group is currently carrying on a cooperative testing program which will provide data from which a method of test for measuring the effectiveness of *transparent* water retardant materials can be developed.

Another task group is studying the problem of water vapor transpiration through water retardant coatings, again with the intention of developing a test method.

Subcommittee VI on Glazed Brick and Tile (E. F. Wanner, chairman) has been quite active during the past year. Recommendations with respect to revisions of Tentative Specifications for Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126 – 58 T) have been accepted by the Administrative Committee on Standards.

The subcommittee is currently studying additional proposed revisions to this tentative specification which, if adopted, will result in more stringent requirements with respect to quality.

Subcommittee VIII on Clay Filter Block (E. F. Wanner, chairman).—There are developments in the trickling filter industry which will require that this subcommittee in the near future consider proposals which will up-date the present Standard Specifications for Vitrified Clay Filter Block for Trickling Filters (C 159 – 55). These changes will involve the addition of requirements for so-called "High-Rate" filter units to this standard.

Subcommittee IX on Chemical Resistant Units (V. W. Weidman, chairman) has devoted much of its time to the development of a specification for ceramic tower packings. Emphasis has been given to a study of the strengths and methods of determining the strength of various materials and shapes of ceramic tower packings. A round-robin test program is being organized. It is hoped that the data resulting from these tests will make it possible to develop a standard method of test and to determine limits for the strength of such ceramic tower packings.

Subcommittee X on Clay Drain Tile (J. G. Sutton, chairman) completed the development of the proposed Tentative Specifications for Clay Drain Tile referred to earlier in the report. This new tentative will replace the present specifications C 4 – 55 and will carry the same designation.

Subcommittee XI, Editorial (Norman Kelch, chairman).—Since the subcommittee has been given new assignments by the Executive Subcommittee, the Editorial Subcommittee should find its work load considerably increased.

This report has been submitted to letter ballot of the committee, which consists of 68 members; 52 members have returned their ballots, of whom 49 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. W. WHITTEMORE,
Chairman.

M. H. ALLEN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126 – 58 T).

Revision and Reversion to Tentative of Standard Specifications for:

Vitrified Clay Filter Blocks for Trickling Filters (C 159 – 55).

Tentative Revision of Standard Methods of Sampling and Testing:

Brick (C 67 – 57).

Tentative Revision of Standard Specifications for:

Building Brick (C 62 – 58), and
Facing Brick (C 216 – 57).

These recommendations were accepted by the Standards Committee on November 6, 1959, and the revised tentative specifications and the tentative revisions of standards appear in the 1959 Supplement to Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-16
ON
THERMAL INSULATING MATERIALS*

Committee C-16 on Thermal Insulating Materials and its subcommittees held two meetings during the year: at Madison, Wis., on October 13, 14, and 15, 1958, and at Pittsburgh, Pa., during ASTM Committee Week, February 1, 2, 3, and 4, 1959. At Madison, the committee was privileged to tour the U. S. Forest Products Laboratories.

At the Madison meeting the committee accepted the resignation of the chairman, W. L. Gantz, because of a change of employment. New officers were elected by the Executive Subcommittee as follows:

Chairman, W. C. Lewis.
First Vice-Chairman, J. R. Allen.
Assistant Secretary, J. R. Bridgeman.

The committee consists of 97 voting members, of whom 50 are classified as producers, 27 as consumers, and 20 as general interest members. There are also 23 nonvoting members, 9 associate members, and 5 honorary members.

At the Pittsburgh meeting Mr. L. Teesdale, formerly with the U. S. Forest Products Laboratories, was elected to honorary membership.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Amosite Asbestos Thermal Insulation for Pipes (C 391 - 59 T) (Accepted April 13, 1959).

Tentative Revision of Standard:

Definitions of Terms Relating to Thermal Insulating Materials (C 168 - 56) (Accepted January 15, 1959).

The new tentative and the tentative revision of standard will appear in the 1959 Supplement to Book of ASTM Standards, Part 5.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods of test as appended hereto:[1]

Tentative Methods of Test for:

Normal Total Emittance of Surfaces of Thin Materials 0.01 in. or Less in Thickness at Approximately Room Temperature,

Determining the Maximum Use Temperature of Preformed High Temperature Insulation, and

Breaking Strength and Calculated Modulus of Rupture of Preformed Thermal Insulation for Pipes.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Diatomaceous Earth Block-Type Thermal Insulation (C 333 - 54 T):[2] Revise as appended hereto.[3]

Tentative Specifications for Diatomaceous Earth Thermal Insulation for Pipes (C 334 - 54 T):[2] Revise as appended hereto.[3]

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

[1] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 5.

[2] 1958 Book of ASTM Standards, Part 5.

[3] The revised tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 5.

Tentative Methods of Test for Water Vapor Transmission of Materials Used in Building Construction (C 355 – 54 T):[2]

Section 3.—In Paragraph (*b*) change the last sentence to read: "Since the permeance of a specimen is generally a function of relative humidity and to a lesser extent temperature, the test conditions must be stated."

In Paragraph (*c*) change the last sentence to read: "Since the permeability of most materials is a function of relative humidity and to a lesser extent temperature, the test conditions must be stated." Add a note to read as follows: "CAUTION.—The definition does not imply that permeance is inversely proportional to thickness when the material is not fully homogeneous."

Section 5(b).—In the Note, change the allowable water gain in silica gel from "2 per cent" to read "4 per cent."

Section 8(d).—Change the allowable water gain in calcium chloride from "6 per cent" to read "10 per cent."

Section 10(b).—In the third sentence following the equation change "1 per cent relative humidity" to read "1 to 3 per cent relative humidity," and change "6 per cent moisture" to read "6 to 10 per cent moisture."

Add the following new Note 3 with regard to the errors in the method:

NOTE 3.—The errors in the method resulting from the tolerances permitted in Section 5 are as follows:

	Possible Error, per cent
Cup ledge (½ in. on 8-in. cup, 1¼-in. thickness)	+12[a]
$CaCl_2$ (10 per cent water, 3 per cent RH)	−6 at 5 perms
Sealant, weight gain or loss	±2
Temperature constant with 1 F	±3[b]
Relative humidity maintained at 50 per cent ±2 per cent	±4[b]
Maximum possible error	+21 or −15

[a] The cup ledge error is negligible for thin materials.

[b] These items are subject to interpretation and not probable.

REVISIONS OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Specifications for 85 per cent Magnesia Thermal Insulating Cement (C 193 – 48):[2] Revise as appended hereto.[4]

Standard Specifications for Expanded or Exfoliated Vermiculite Thermal Insulating Cement (C 196–48):[2] Revise as appended hereto.[4]

Standard Specifications for Diatomaceous Silica Thermal Insulating Cement (C 197 – 48):[2] Revise as appended hereto.[4]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Structural Insulating Board Made from Vegetable Fibers (**C 208 – 57 T**),

Mineral Wool Batt Insulation (Industrial Type) (**C 262 – 55 T**),

Mineral Wool Felt Insulation (Industrial Type) for Low Temperatures (**C 264 – 55 T**),

Mineral Wool Molded-Type Pipe Insulation for Low Temperatures (**C 300 – 52 T**),

Calcium Silicate Block-Type Thermal Insulation (**C 344 – 54 T**),

Calcium Silicate Thermal Insulation for Pipes (**C 345 – 54 T**),

Mineral Wool Block or Board Thermal Insulation for Low Temperatures (**C 378 – 56 T**),

Mineral Wool Felt Insulation (Industrial Type) for Elevated Temperatures (**C 382 – 56 T**), and

Mineral Wool Block or Board Thermal Insulation for Elevated Temperatures (**C 392 – 57 T**).

Tentative Methods of:

Testing Structural Insulating Board Made from Vegetable Fibers (**C 209 – 57 T**),

Test for Thermal Conductance and Transmittance of Built-Up Sections by Means of the Guarded Hot Box (**C 236 – 54 T**),

---

[4] The revised specifications appear in the 1959 Supplement to Book of ASTM Standards, Part 5.

Test for Thermal Conductivity of Pipe Insulation (**C 335 – 54 T**),
Test for Mean Specific Heat of Thermal Insulation (**C 351 – 54 T**),
Test for Linear Shrinkage of Preformed High-Temperature Thermal Insulations Subjected to Soaking Heat (**C 356 – 55 T**),
Sampling Preformed Thermal Insulation (**C 390 – 57 T**), and
Test for Consistency of Wet-Mixed Thermal Insulating Cement (**C 405 – 57 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee S-II on Structural Thermal Insulation (A. W. Chapman, chairman).—The scope of this subcommittee has been amplified to encompass many types of structural insulation other than those included in the original scope of the committee when it was designated "Structural Insulating Board." Fiberboard products are now redesignated for use as other than ordinary sheathing materials and are receiving the attention of this subcommittee. Examples of these are insulating roof deck slabs and insulating formboard. Test methods for both of these are being developed currently.

Subcommittee S-IV on Blanket Insulation (G. E. Elford, chairman) has been working for some time on a specification for blanket thermal insulation for buildings, and a new draft will shortly be submitted to the membership. A separate document covering methods of testing this blanket insulation is also being developed. Revision of specifications covering industrial type mineral wool blanket, batt, and felt thermal insulations is to be undertaken. Existing definitions are being revised and new definitions are being developed for materials handled by the subcommittee.

Subcommittee T-IX on Vapor Transmission (F. A. Joy, chairman).—As a result of tests covering a period of three years, the subcommittee has approved the revisions of Methods C 355 referred to earlier in the report to permit a larger moisture gain in the dry-cup procedure. A round-robin on this test method was conducted by eight participating laboratories designed to show the reliability of the method. Explanation of the possibility of designating a standard water vapor transmission specimen has shown promise, and the work will be expanded. Recommendations for needed research clarifying the behavior of moisture and its control in insulation have been made. A study of good practice recommendations for vapor barrier applications is planned.

This report has been submitted to letter ballot of the committee, which consists of 97 voting members; 83 members returned their ballots, of whom 80 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. C. LEWIS,
Chairman.

J. M. HIGH,
Secretary.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the following recommendation:

Revision of Tentative Method of Test for:

Mean Specific Heat of Thermal Insulation (C 351 – 54 T).

This recommendation was accepted by the Standards Committee on September 15, 1959, and the revised tentative method appears in the 1959 Supplement to Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-17
ON
ASBESTOS-CEMENT PRODUCTS*

Committee C-17 on Asbestos-Cement Products and its subcommittees held one meeting during the year: at Boston, Mass., on June 26, 1958, during the Annual Meeting of the Society.

The committee consists of 29 voting members, of whom 9 are classified as producers, 5 as consumers, and 15 as general interest members.

Liaison with Committee E-6 on Methods of Testing Building Constructions continues, with S. E. Williams representing Committee C-17 when there are problems of mutual interest. Since Committee E-6 is concerned with methods of test in construction practices with special reference to building codes, it will consult with Committee C-17 on specific uses of asbestos-cement.

The officers elected in 1958 for the ensuing term of two years are as follows:

Chairman, W. V. Friedlaender.
Vice-Chairman, C. R. Hutchcroft.
Secretary, C. C. Kelsey.

The committee regrets to report the death of C. C. Kelsey, who had been secretary of the committee for many years. Following his death, S. Collier was appointed to fill the position.

The American Standards Assn. has accepted participating membership in Committee 77 on Asbestos-Cement Products of the International Organization for Standardization (ISO). Committee C-17 is handling United States participation in this ISO work.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee C-17 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications and Methods of Test for:

Asbestos-Cement Non-Pressure Sewer Pipe (C 428 – 59 T).

Tentative Revision of Standard Specifications and Methods of Test for:

Flat Asbestos-Cement Sheets (C 220 – 55),
Asbestos-Cement Roofing Shingles (C 222 – 55), and
Asbestos-Cement Siding Shingles and Clapboards (C 223 – 55).

Revision and Reversion to Tentative of Standard Specifications and Methods of Test for:

Corrugated Asbestos-Cement Sheets (C 221 – 55), and
Asbestos-Cement Pressure Pipe (C 296 – 55).

The recommendations concerning Standard Specifications and Methods C 220 – 55, C 221 – 55, C 222 – 55, and C 223 – 55 were accepted by the Standards Committee on September 9, 1958, and the tentative revisions appear in the 1958 Book of ASTM Standards, Part 5. The recommendations concerning Specifications and Methods C 296 – 55 and C 428 – 59 T were accepted on February 9, 1959, and the new and revised specifications and methods of test appear

---

*** Sixty-second Annual Meeting of the Society, June 21–26, 1959.**

in the 1959 Supplement to Book of ASTM Standards, Part 5.

In approving the new Tentative Specifications and Methods of Test for Asbestos-Cement Non-Pressure Sewer Pipe (C 428 – 59 T) and the revision of Standard Specifications and Methods of Test for Asbestos-Cement Pressure Pipe (C 296 – 55), the note to the section on inspection and rejection was revised to read as follows:

NOTE.—To insure that pipe furnished under these specifications conforms to the requirements, and in order that the purchaser can conveniently arrange for the witnessing of the tests, the purchaser may require such tests to be conducted in the country where the product is installed. Such tests, in accordance with these specifications, testing facilities, and expense thereof, shall be matters of agreement between the supplier and purchaser.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Research (William Lerch, chairman).—A progress report on the study of determination of organic content in asbestos-cement products will be sent to each member of the committee, and the subcommittee will investigate further the effect of coloring materials, coatings, and carbon black. The subcommittee is working on a definition of completeness of cure, approaching the problem by studying heat of hydration tests.

Task Force on Nomenclature (W. A. Heausler, chairman).—A list of terms and definitions has been submitted to the task force for comments and suggestions.

This report has been submitted to letter ballot of the committee, which consists of 30 members; 20 members returned their ballots, of whom 19 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

W. V. FRIEDLAENDER,
Chairman.

S. COLLIER,
Secretary.

REPORT OF COMMITTEE C-19
ON
STRUCTURAL SANDWICH CONSTRUCTIONS*

Committee C-19 on Structural Sandwich Constructions held three meetings during the year: on June 24 and 25, 1958, in Boston, Mass., in conjunction with the Annual Meeting of the Society; on October 21 and 22, 1958, at the Martin Co., in Baltimore, Md.; and on April 21 and 22, 1959, at Chance Vought Aircraft, in Dallas, Tex. An Executive Subcommittee meeting was held on August 12, 1958, at Society Headquarters in Philadelphia, Pa.

The committee consists of 72 voting members, of whom 39 are classified as producers, 9 as consumers, and 24 as general interest members.

Three Advisory Committee members-at-large were appointed: T. J. Martin, K. D. Rider, and J. M. Stevens.

A Symposium on Durability and Weathering of Structural Sandwich Constructions,[1] sponsored by Committee C-19, is being planned for presentation at the Third Pacific Area National Meeting in San Francisco, Calif., on October 13, 1959. Seven papers comprising two sessions are planned.

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Mechanical Properties of Basic Materials (Steve Yurenka, chairman) is preparing tentative methods for determining fluid flow in honeycomb cores, and for impact resistance of sandwich core materials. Existing standards and tentatives have been reviewed, and several minor revisions are under consideration.

Subcommittee II on Mechanical Properties of Basic Sandwich Construction (E. W. Kuenzi, chairman).—Comments received as a result of a ballot on two proposed tentatives on flexure-creep tests and peel tests of sandwich construction are being reviewed. The climbing drum peel test developed by Committee D-14 on Adhesives has been recommended for use by the subcommittee. Recommendations for elevated temperature testing are also being developed.

Subcommittee III on Permanence, Durability, and Simulated Service (L. Johnson, chairman).—The "Alpha" exposure program begun in 1955 was completed in the spring of 1958. One participant is reporting results of his portion in the symposium mentioned previously. The "Beta" program was completed in the spring of 1959. A third program, "Gamma," was initiated in April, 1959, in which 108 panels of 9 different commercial types are being exposed. A special task group is considering temperature cycles for a proposed laboratory aging test.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The papers in this symposium will be published as a Special Technical Publication *ASTM STP No. 270*.

Subcommittee IV on Nomenclature and Definitions (R. A. Biggs, chairman) is compiling new definitions and terms for consideration.

Respectfully submitted on behalf of the committee,

THEODORE PAJAK,
Chairman.

J. H. GIBBUD,
Secretary.

REPORT OF COMMITTEE C-20
ON
ACOUSTICAL MATERIALS*

Committee C-20 on Acoustical Materials held two meetings during the year: at Philadelphia, Pa., September 25, 1958; and on April 16, 1959 in Chicago, Ill.

The committee consists of 66 members, of whom 24 are classified as producers, 11 as consumers, and 31 as general interest members. There is 1 consulting member.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sound Absorption (H. J. Sabine, chairman).—The scope of this subcommittee has been expanded to include sound transmission. The subcommittee will review and consider for adoption the method now being developed for the Acoustical Materials Assn. which evaluates sound transmission between adjacent rooms, the sound finding a path up through the first acoustical ceiling, along the space behind, over the part-high partition, and down through the second acoustical ceiling. Work continues on revision of Method of Test for Sound Absorption of Acoustical Materials in Reverberation Rooms (C 423 – 58 T). Work continues on evaluation of three small scale methods of measuring sound absorption: use of horn coupler, box method, shallow duct method.

Subcommittee II on Flame Resistance (Wallace Waterfall, chairman).—The correlation tests by laboratories employing the tunnel method for flame spread rating of acoustical materials by the Method of Fire Hazard Classification of Building Materials (E 84 – 50 T) were completed. In the opinion of the subcommittee, the correlations between laboratories were unsatisfactory. The report has been made available to Committee E-5 with the request that it consider making appropriate changes in Method E 84 and inform Committee C-20 of the proposed changes. The subcommittee recommended that Committee E-5 conduct correlation tests after known variations between tunnels have been corrected. The subcommittee believes that better calibration of radiometers will improve correlations between laboratories using the radiant panel method.

Subcommittee III on Maintenance (W. M. Rees, chairman).—Studies on causes of soiling of acoustical materials conducted at the National Bureau of Standards under sponsorship of the Public Buildings Administration and the Acoustical Materials Assn. have been followed by the subcommittee. The work has been discontinued without reaching specific conclusions. Present subcommittee activity is aimed at developing a standard procedure for repainting materials and evaluating their subsequent sound absorption.

Subcommittee IV on Application (L. F. Yerges, chairman).—Strength and space problems of mechanical suspension systems are being considered. A questionnaire will be circulated to gather information on locations of failures in suspension systems. Corrosion problems caused by indoor operation under the influence of air conditioning factors will be studied.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

Subcommittee V on Basic Physical Properties (W. A. Jack, chairman).—Correlation tests on measurement of light reflectance is being continued, using a set of plaques furnished by the National Bureau of Standards. Participating laboratories are using sphere reflectometers and various modifications of 0- to 45-deg instruments. The Acoustical Materials Assn. continues to use a 10-in. sphere reflectometer. The draft of methods for measuring flow resistance of acoustical materials is being prepared. Activities in sound transmission have been transferred to Subcommittee I.

This report has been submitted to letter ballot of the committee, which consists of 66 voting members; 22 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. N. HAMME,
Chairman.

RALPH HUNTLEY,
Secretary.

REPORT OF COMMITTEE C-21
ON
CERAMIC WHITEWARES AND RELATED PRODUCTS*

Committee C-21 on Ceramic Whitewares and Related Products held two meetings during the year: in Bedford Springs, Pa., on September 25, 1958, and in Chicago, Ill., on May 18, 1959.

The committee consists of 81 voting members, of whom 40 are classified as producers, 13 as consumers, and 28 as general interest members.

During the course of the year, Task Group A on Nonmetallic Magnetic Materials attained subcommittee status as Subcommittee VII on Nonmetallic Magnetic Materials.

A task group formed for the purpose of establishing tests for detergent resistance of decals was absorbed into Subcommittee III on Fundamental Properties. A task group was formed under the chairmanship of A. J. Metzger to deal with foamed ceramics as radome materials. A task group designed to deal with the problem of tensile strength determination was formed as the outgrowth of a letter ballot on a proposed method of test.

Subcommittee Activities

Subcommittee I on Nomenclature (A. S. Watts, chairman) is working on improvement of many standard definitions and has asked for committee-wide balloting on several of these, including revisions of Rockingham ware, glazed extra-duty tile, faience, and majolica.

Subcommittee II, Editorial (Van E. Campbell, chairman) has reviewed Definitions of Terms Relating to Ceramic Whitewares and Related Products (C 242 – 58 T) and sent suggestions for revision to Subcommittee I on Nomenclature. Under consideration is a compilation of standards prepared by Committee C-21.

Subcommittee III on Fundamental Properties (W. C. Mohr, chairman) continued work on methods to determine translucency of fired whiteware materials, lead solubility of glazes, and tensile strength. A task group was formed to investigate methods of test for alkali-resistance of glazes; an alternative method is now under consideration. A proposed method for absorption of liquid by fired whiteware materials has been written and interlaboratory testing started. Impact testing is being reviewed.

Subcommittee IV on Clays (G. W. Phelps, chairman).—Work on a hydrometer technique for particle-size determination of clays has reached a point of refinement where a method will be submitted to subcommittee ballot. A companion procedure for particle-size determination in the minus 2-micron range using the centrifuge has been written and is undergoing revision by the subcommittee, as are tests for organic constituents and soluble sulfate content of whiteware clays. Plans are under way for extending modifications of the particle-size techniques to nonplastic whiteware materials.

Subcommittee V on Nuclear Applications (John M. Warde, chairman) is actively working on specifications for reactor grade graphite. Joseph Handwerk has assumed the chairmanship for 1959 and is organizing a symposium in the field of ceramics for nuclear applications for presentation at the Pacific Coast meeting of ASTM in October, 1959.[1]

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

Subcommittee VI on Ceramic Tile (J. V. Fitzgerald, chairman) is actively developing 21 methods of test for tile. Recommendations were made to Subcommittee I for clarifying certain terms relating to tile.

Subcommittee VII on Non-Metallic Magnetic Materials (Horst Kedesdy, chairman).—A proposed method of test for ferrimagnetic resonance linewidth and gyromagnetic ratio of non-metallic magnetic materials and a proposed method of test for complex dielectric constant of non-metallic magnetic materials have been passed upon by a group headed by S. L. Blum and by the subcommittee. A third proposed method of test written by a group headed by Melvin Eisenberg is still under discussion. It is proposed to discuss tests dealing with Curie temperature, saturation magnetization, and B-H curves during the Annual Meeting of the Society. Melvin Eisenberg is now secretary of this subcommittee.

This report has been submitted to letter ballot of the committee, which consists of 81 voting members; 49 members returned their ballots, of whom 46 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

M. D. BURDICK,
Chairman.

G. W. Phelps,
Secretary.

---

[1] The papers presented at this symposium are to be published as a Special Technical Publication *ASTM STP No. 276.*

REPORT OF COMMITTEE C-22

ON

PORCELAIN ENAMEL*

Committee C-22 on Porcelain Enamel held two meetings during the year: on October 2 and 3, 1958, in Annapolis, Md., as the guests of the U. S. Naval Engineering Experiment Station; and on February 5 and 6, 1959, in Pittsburgh, Pa., during ASTM Committee Week.

The committee consists of 48 members of whom 42 are voting members; 22 are classified as producers, 5 as consumers, and 15 as general interest members.

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Abrasion Resistance of Porcelain Enamels as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for 45-deg Specular Gloss of Ceramic Materials (**C 346 – 55 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as follows of the Standard Method of Test for Adherence of Porcelain Enamel and Ceramic Coatings to Sheet Metal (**C 313 – 55**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society.

Section 1.—Revise the Note at the end of the section to read as follows:

> **NOTE.—This method is not applicable to metals over 0.082 in. or under 0.016 in. in thickness, nor to porcelain enamels on aluminum or aluminum alloys. Some anomalies have also been noted in certain processes in which the cover coat is applied directly to sheet steel. Some processes used in applying the cover coat directly to sheet steel might require a different rating criterion than is used in evaluating conventional ground coats on steel.**

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

Impact Resistance of Porcelain Enameled Utensils (**C 284 – 51 T**),
Warpage of Porcelain Enameled Flatware (**C 314 – 53 T**),
Fusion Flow of Porcelain Enamel Frits (Flow-Button Methods) (**C 374 – 55 T**), and
Torsion Resistance of Laboratory Specimens of Porcelain Enameled Iron and Steel (**C 409 – 57 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

*** Sixty-second Annual Meeting of the Society, June 21–26, 1959.**

[1] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 5.

[2] 1958 Book of ASTM Standards, Part 5.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (B. J. Sweo, chairman) is continuing its work on the evaluation of porcelain enamel and ceramic coatings on high- and low-alloy steels at elevated temperatures and its survey of engineering data relating to the enamel-metal system. The subcommittee is initiating a program for the promotion of research concerned with porcelain enamel problems and has developed a list of some 16 topics which are being given detailed consideration.

Subcommittee II on Nomenclature (E. E. Howe, chairman) has continued its work on definitions of terms used in the porcelain enamel industry.

Subcommittee III on Education (L. S. O'Bannon, chairman) has continued its contacts with the technical press and informing interested companies of the work of the committee.

Subcommittee IV on Materials (H. S. Saunders, chairman) has six active projects and is responsible for the recommendations concerning Methods C 374 and C 409 as noted earlier in this report.

Subcommittee V on Finished Products (J. C. Richmond, chairman) has 11 projects measuring physical properties of enamels. It recently completed (1) the proposed Tentative Method of Test for Abrasion Resistance of Porcelain Enamels; (2) revision of the Standard Method of Test for Adherence of Porcelain Enamel and Ceramic Coatings to Sheet Metal (C 313 – 55); and (3) the recommendations concerning Methods C 284, C 314, and C 346 as noted earlier in this report.

This report has been submitted to letter ballot of the committee, which consists of 42 voting members; 34 members returned their letter ballots, of whom 31 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. N. HARRISON,
Chairman.

G. H. SPENCER-STRONG,
Secretary.

REPORT OF COMMITTEE D-1
ON
PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS*

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: on June 23 to 25, 1958, in Boston, Mass., in connection with the Annual Meeting of the Society, and in Columbus, Ohio on January 19 to 21, 1959.

At the June, 1958, meeting, W. K. Asbeck, Research Department, Union Carbide Chemicals Co., South Charleston, W. Va., presented a most stimulating paper on "The Adhesion of Organic Coatings to Various Substrates." This paper covered the theory and measurement of the factors which cause coatings to adhere to metal, wood, and plastic and the effects of inter-coat adhesion on the over-all adhesion of multiple-coat systems.

At the January, 1959, meeting, E. G. Bobalek, Professor of Chemical Engineering, The Case Institute of Technology, Cleveland, Ohio, presented an illustrated talk on "Film Stabilization in Paint Testing." This paper first pointed out the importance of the circumstances prevailing during the process of film formation, then stated and illustrated the probability of the occurrence of the preferred steady state conditions under service conditions of paint application, and finally discussed several alternatives for conducting more significant investigations to predict service performance of paints.

Committee D-1 has continued its close cooperation with the Federation of Paint and Varnish Production Clubs. Ninety-six standards have been jointly approved by both organizations. The Joint Federation-ASTM Committee on Paint, Varnish, and Lacquer (J. C. Weaver, chairman) serves as the permanent liaison between the two organizations.

The American Standards Assn. has approved as American Standard during the year the following ASTM specifications which are under the jurisdiction of Committee D-1:

Specifications for Raw Linseed Oil (ASTM D 234 - 58; ASA K34.1—1959), and
Specifications for Boiled Linseed Oil (ASTM D 260 - 58; ASA K35.1—1959).

The Committee on Inter-Committee Relations (E. W. Fasig, chairman) is composed of the official representatives of Committee D-1 on other ASTM technical committees. The committee thus serves to maintain close contact between Committee D-1 and those committees whose activities are of interest to Committee D-1. In the past year, representatives of Committee D-1 have been appointed to the new Committees D-26 on Halogenated Organic Solvents, and F-2 on Flexible Barrier Materials.

During the year, Subcommittee III on Bituminous Emulsions (R. H. Cubberley, chairman) completed its assignment and was discharged upon its own request, with Mr. Cubberley named as advisor.

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

NEW STANDARDS

The methods listed below currently appear in the Standard Methods of Testing Varnishes (**D 154 - 58**).[1] The committee recommends that these methods be published as separate standards. The sections of Standard Methods D 154 - 58 where these tests currently appear are indicated; the methods themselves are not appended to this report. In several cases it has been necessary to make minor changes in the methods as indicated in order to complete them for publication as separate standards.

Standard Methods of Test for Nonvolatile Content of Varnishes (Present Sections 14 to 17 of Methods D 154 - 58):

New Section.—Add the following new section on scope:

Scope.—This method determines the fraction of a varnish which is relatively stable at the temperature of the test while volatile solvents are driven off. It is sometimes an approximate measure of the film-forming matter in a varnish.

Standard Methods of Test for Elasticity or Toughness of Varnishes (Present Sections 20 to 32 of Methods D 154 - 58):

New Section.—Add the following new section on scope:

Scope.—These methods determine elasticity or toughness of any varnish which is miscible with the diluents specified, under the conditions of the test.

The Section on Procedure, under Method B, has been editorially revised.

Standard Method of Test for Acid Value of Varnishes (Present Sections 33 to 35 of Methods D 154 - 58):

New Section.—Add the following new section on scope:

Scope.—This method determines acid value of varnishes and paint liquids containing normally small percentages of driers or other soluble metal soaps. Larger amounts of soluble metals may cause repeated fading of the end points of titrations and abnormally high results which can be mitigated by the method of Diener and Werthen.

Standard Method of Test for Reactivity of Paint Liquids (Present Sections 36 to 40 of Methods D 154 - 58).

Standard Method of Test for Resistance to Water and Alkali of Dried Films of Varnishes (Present Sections 41 to 46 of Methods D 154 - 58):

New Sections.—Add the following new sections on scope and apparatus:

Scope.—This method determines the resistance of a dried varnish film to water.

Apparatus.—(*a*) Beaker, glass, 600 ml or larger.

(*b*) Tin-plate panels, 3 by 5 in., cut from commercial No. 31 gage bright tin plate, weighing 0.39 to 0.51 lb per sq ft and carefully cleaned with toluene and dried before use.

Procedure.—Change the first sentence of the Section on Procedure covering the Test for Resistance of Dried Films to Water to read: "Pour the varnish on a tin panel, allow to drain in a nearly vertical position and allow to dry for 48 hr at 25 ± 2 C and 50 ± 10 per cent relative humidity.

Standard Methods of Test for Drying Times of Varnishes (Present Sections 47 to 55 of Methods D 154 - 58).

Standard Method of Test for Exterior Durability of Varnishes (Present Sections 56 to 60 of Methods D 154 - 58).

NEW TENTATIVES

The committee recommends for publication as tentative the following seven specifications and methods as appended hereto:[2]

---

[1] 1958 Book of ASTM Standards, Part 8.

Tentative Specifications for:

Strontium Chromate, and
Basic Lead Silico-Chromate.

Tentative Methods of Test for:

Phthalic Acid Isomers and Benzoic Acid in Alkyd Resins and Esters,
Epoxy Content of Epoxy Resins, and
Moisture Vapor Permeability of Organic Coating Films.

Tentative Method for:

Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments, and
Sampling and Testing Shellac Varnish.

The following two methods currently appear in the Tentative Methods of Testing Varnishes (**D 154 – 58 T**),[1] and the committee recommends that they be published as separate tentatives. The sections of Tentative Methods D 154 – 58 T where these tests currently appear are indicated; the methods are not appended to this report. Several minor changes are recommended as indicated.

Method of Test for Gas Checking and Draft Test of Varnish Films (Present Sections 2 to 14 of Tentative Methods D 154 – 58 T):

Sections 4(f) and 7(e).—Change to read: "Tin-Plate Panels, 3 by 5 in. or smaller, cut from Commercial No. 31 gage bright tin plate, weighing 0.39 to 0.51 lb per sq ft and carefully cleaned with toluene and dried before use."

Section 12.—Change to read as follows:

12. *Test Conditions.*—Perform the draft test preferably at 25 ± 2.2 C (77 ± 4 F) and 50 ± 4 per cent RH. When these conditions cannot be obtained or maintained for the duration of the test, include a record of the temperature and humidity range in the report.

Method of Test for Print Resistance of Varnishes (Present Sections 18 to 23 of Tentative Methods D 154 – 58 T):

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentative specification and methods and their continuation as tentative:

Tentative Specifications for Heavy Petroleum Spirits (Heavy Mineral Spirits) (D 965 – 48 T):[1]

Section 3(c).—Change to read as follows:

(*c*) *Blackening.*—Method of Test for Copper Corrosion by Mineral Spirits (Copper Strip Test) ASTM Designation: D 1616). Class 1 and Class 2 strips are free from blackening and are considered passing. Class 3 and Class 4 strips do not pass.

Tentative Method of Test for Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus (D 1310 – 56 T):[1]

Section 3(c).—Change the last sentence to read: "For flash points from 20 to 40 F, use ASTM Tag Closed Tester, Low Range, Thermometer 57F. For flash points below 20 F, use ASTM Thermometer 33F."

Note 2.—Change to read: "The original Tag Open-Cup (Paper Scale) Thermometer will be a permissible alternate until January 1, 1962."

Tentative Methods of Testing Fatty Acids Used in Protective Coatings (D 1467 – 57 T):[1] Revise as appended hereto.[3]

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revision as follows of the Standard

---

[2] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 8.

[3] The revised tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 8.

Definitions of Terms Relating to Paint, Varnish, Lacquer and Related Products (D 16 – 58):[1]

Add the following 12 new definitions:

Chromaticity Coordinates.—The ratios of each of the tristimulus values of a color to the sum of the tristimulus values. (Chromaticity coordinates in the CIE system of a color specification are designated by x, y, and z.)

Color Difference.—The magnitude and character of the difference between two object colors under specified conditions.

Distinctness-of-Image Gloss.—The sharpness with which image outlines are reflected by the surface of an object.

Gloss.—The aspect of the reflecting properties of an object determined by the brightness and configuration of images.

Metamers.—Specimens which color match when observed in light of one spectral composition but which do not match in light of some other spectral composition.

Munsell Color System.—A system for color specification of surfaces illuminated by daylight and viewed by an observer adapted to daylight. The system yields approximations to scales of the variables, hue, saturation, and lightness, with uniform perceptual spacing. In this system these variables are designated hue, chroma, and value, respectively.

Munsell Chroma.—Index of the saturation of the perceived color of an object as described in the Munsell Color System.

Munsell Hue.—Index of the hue of the perceived color of an object as described in the Munsell Color System.

Munsell Value.—Index of the lightness of the perceived color of an object as described in the Munsell Color System.

Sheen.—Shiny or lustrous appearance at near grazing incidence of a surface that appears mat for near perpendicular incidence; also, the 85-deg specular gloss of such a surface.

Specular Gloss.—The surface reflectance of an object evaluated for specified angle of unidirectional illumination when viewed at the angle of mirror reflectance by a receptor of specified aperture.

Tristimulus Values.—The amounts of each of three primaries in additive mixture required to match a color. (Tristimulus values in the CIE system of color specification are designated by X, Y, and Z.)

REVISION OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following five standards and their reversion to tentative:

Standard Specifications for Raw Tung Oil (D 12 – 55):[1]

Section 2.—Add the following requirement:

Flash point, min.................. 250 F

Section 4.—Add the sentence: "Flash point shall be determined in accordance with the Method of Test for Flash Point of Drying Oils (ASTM Designation: D 1393)."

Standard Methods of Sampling and Analysis of Shellac (D 29 – 57):[1] Revise as appended hereto.[4]

Standard Methods of Testing Varnishes (D 154 – 58):[1] Revise as appended hereto.[4]

Standard Methods of Sampling and Testing Aluminum Powder and Paste (D 480 – 51):[1] Revise as appended hereto.[4]

Standard Specifications for Liquid Paint Driers (D 600 – 43):[1] Revise as appended hereto.[4]

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

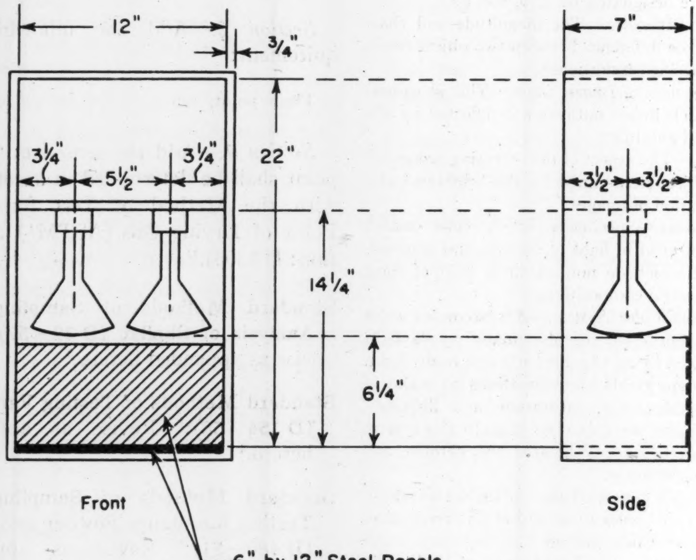
The committee recommends that the Tentative Method of Test for Flash Point of Drying Oils (**D 1393 – 56 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

---

[4] The revised specifications and methods appear in the 1959 Supplement to Book of ASTM Standards, Part 8.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following five standards as indicated, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:



Two G. E. reflector infrared industrial 250-w lamps.
Two stainless steel panels, timer, powerstat, aluminum foil disher (diameter 58 mm, height 18 mm), and analytical balance (accurate to 0.001 g).

FIG. 1.—Infrared Oven.

Definitions of Terms Relating to Paint, Varnish, Lacquer and Related Products (D 16 – 58):[1]

Replace the definition of Color with the following four new definitions:

Color of an Object.—The aspect of the appearance of an object dependent upon the spectral composition of the incident light, the spectral reflectance or transmittance of the object, and the spectral response of the observer.

Hue.—The attribute by which a perceived color is distinguished as red, yellow, green, blue, purple, or a combination of these. (White, gray, and black colors possess no hue.)

Lightness.—The attribute by which a perceived color is judged to be equivalent to a member of the continuous series of grays ranging from black to white.

Saturation.—The attribute by which a perceived color is judged to depart from gray of equal lightness toward a pure hue.

Specifications for Petroleum Spirits (Mineral Spirits) (D 235 – 39):[1]

Section 3(c).—Change to read as indicated above for Section 3(*c*) of Tentative Specifications D 965.

Specifications for Isopropyl Alcohol (Isopropanol) (D 770 – 58):[1]

Section 2.—Change the last four lines to read as follows:

Water miscibility...	miscible without turbidity when diluted to 10 volumes with distilled water at 25 C.

Methods of Chemical Analysis of White Linseed Oil Paints (D 215 - 41):[1]

Section 5.—Change to read as follows:

5. *Volatile Thinner.*—Using a 10-ml Luer syringe, weigh accurately (±0.001 g) from 0.4 to 0.6 g of the sample in duplicate into tared aluminum foil dishes (58 mm diameter, 18 mm high) spreading the paint over the bottom of the dish. The weighing should be completed quickly to prevent loss of volatile matter while weighing. Add 1 ml of toluene, shake to disperse, and heat for 20 min in the infrared oven constructed as shown in Fig. 1. Cool in desiccator and weigh. Calculate the loss in weight as percentage of water and volatile thinner. Subtract from this the percentage of water as determined according to the Method of Test for Water in Petroleum Products and Other Bituminous Materials (ASTM Designation: D 95). Report the remainder as per cent volatile thinner.

Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 - 58):[1]

Sections 20, 21, and 22.—Delete these sections which describe the test for free phenols in tricresyl phosphate, since this property is no longer a part of the Specifications for Tricresyl Phosphate (D 363 - 56).

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Methods of Testing Varnishes (**D 154 - 58 T**)[1] because the various tests covered are now being issued under separate designations as recommended earlier in this report.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood (**D 1211 - 57 T**).[1]

APPROVAL OF REVISION OF STANDARD

The proposed revisions in the specifications for ASTM Thermometers No. 88C and 88F, and No. 9F and 57F, of the Standard Specifications for ASTM Thermometers (E 1 - 58), developed by Committee E-1 on Methods of Testing, are recommended for approval. Thermometers No. 88C and 88F are specified in Tentative Method of Test for Flash Point of Drying Oils (D 1393 - 56 T) which is under the jurisdiction of Committee D-1, and Thermometers No. 9F and 57F are specified in Standard Method of Test for Flash Point by Tag Closed Tester (D 56 - 56), which is under the joint jurisdiction of Committee D-1 and Committee D-2 on Petroleum Products and Lubricants.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (D. S. Bolley, chairman) prepared the revision of the Methods of Testing Fatty Acids (D 1467 - 57 T) which consists of three additional methods of test for ash, rosin acids, and color change on heating. The subcommittee also prepared the proposed revision of the Specifications for Raw Tung Oil (D 12 - 55) with the recommendation that it revert to tentative. In cooperation with the Joint Liaison Group on Fatty Acids, the subcommittee is continuing to work on the development of specifications for tall oil fatty acids, on a method of test for hydroxyl value, and on an isomerization procedure. In addition to the proposed revision of the Specifications for Raw Tung Oil (D 12) which calls for the inclusion of a minimum flash point requirement, the subcommittee is completing its study looking toward the revision of the procedure for gel time of tung oil and its removal from the Standard Methods of Testing Drying Oils (D 555 – 58) for publication as a separate method of test. The subcommittee has undertaken a thoroughgoing revision of Methods D 555 with the purpose of removing the individual procedures and establishing each method of test under its own numerical designation. Method D 555 will then remain as an index and description of the significance of the tests applicable to drying oils. The subcommittee is continuing its study of a procedure for determining the presence of fish oil in drying oils.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on Bituminous Emulsions (R. H. Cubberley, chairman), having completed its assignment and having no plans for further activity, is being discharged.

Subcommittee IV on Traffic Paint (W. W. Burr, chairman) is continuing its development of both accelerated tests and road tests for measuring the performance properties of traffic paints. It is cooperating with Subcommittee XVIII on Physical Properties of Materials in the development of a method of test for weight per gallon of paint containing glass beads, pumice, or other coarse particles.

Subcommittee V on Solvents (E. F. Rogers, chairman), prepared the proposed revisions of the Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 – 58), Specifications for Petroleum Spirits (D 235 – 39), Specifications for Isopropyl Alcohol (D 770 – 58), and of Specifications for Heavy Petroleum Spirits (D 965 – 48 T). The subcommittee is completing work on specifications for isobutyl alcohol and isobutyl acetate, and on methods of test for determination of water solubility of acetone, water solubility of isopropyl alcohol, and permanganate time of tricresyl phosphate. It is continuing work on the revision of the Method of Test for Aniline Point and Mixed Aniline Point of Hydrocarbon Solvents (D 1012 – 51), Test for Kauri-Butanol Value of Hydrocarbon Solvents (D 1133 – 54 T), and Specifications for Methyl Ethyl Ketone (D 740 – 56). It is proceeding with its work to consolidate into a single method of test the three separate procedures for Toluene Dilution Ratio of the Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 – 58), Toluene Dilution of Specifications and Methods of Test for Soluble Cellulose Nitrate (D 301 – 56), and the Method of Test for Nitrocellulose Diluting Power of Hydrocarbon Solvents (D 1134 – 53). It has begun the development of an improved description and determination of the solvency of mineral spirits and is continuing its cooperation with Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials on the revision of the specifications for industrial grade benzene, toluene, and xylene. It has established a new working group on chemical intermediates which has begun the development of methods of test and specifications for formaldehyde and for vinyl acetate.

Subcommittee VI on Definitions (F. B. Stieg, Jr., chairman), in cooperation with Subcommittee X on Optical Properties, prepared the definitions of sixteen optical terms, four of which are a replacement for the present definition of Color in the Standard Definitions of Terms D 16 – 58, and the other twelve are to be added as a tentative revision of the standard. The subcommittee has undertaken the preparation of definitions of the terms "build" and "durability."

Subcommittee VII on Accelerated Tests for Protective Coatings (N. B.

Garlock, chairman) prepared the proposed Tentative Method of Evaluation of Painted Specimens Subjected to Corrosive Environments. The subcommittee is completing work on a method of test for color change of white architectural enamels, methods for the preparation of tin-plate panels and of aluminum panels, and a water-fog test in addition to a proposed revision of the Standard Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer and Related Products (D 609 - 52). It is actively working on a method for the preparation of concrete and masonry test panels, on methods of test for perspiration resistance and for detergent resistance, and on a cold-crack test method. It is also studying tests for resistance of house paint to blistering, and methods of evaluating test panels after water exposure.

Subcommittee VIII on Methods of Chemical Analysis of Paint Materials (W. H. Madson, chairman) prepared the proposed revision of the Standard Methods for Chemical Analysis of White Linseed Oil Paints (D 215 - 41). It is continuing the development of a method for the determination of water-soluble salts of iron oxide pigments by the specific resistance method and has undertaken to adapt the method to other pigments. It is engaged in the revision of the Tentative Methods for Chemical Analysis of White Titanium Pigments (D 1394 - 56 T), and the development of methods for the analysis of strontium chromate pigments and the determination of lead at low concentrations in paint.

Subcommittee IX on Varnish (J. C. Weaver, chairman) prepared the thoroughgoing revision of the Standard Methods of Testing Varnishes (D 154 - 58) which is proposed for reversion to tentative. The revision of Methods D 154 has involved first of all the removal from it of nine procedures which are proposed as separate standards. That part of Methods D 154 which remained after removal of the nine procedures has been rewritten as a general guide to the testing of varnishes and as an index to the methods applicable to varnish testing. Of the nine procedures mentioned, the following six are proposed for immediate adoption as standard methods of test: nonvolatile content of varnishes, elasticity or toughness of varnishes, acid value of varnishes, drying time of varnishes, resistance to water and alkali of dried films of varnishes, and exterior durability of varnishes. A seventh procedure, for reactivity of paint liquids, is recommended for immediate adoption as standard with the ASTM designation D 479 under which it was originally published prior to having been incorporated into Methods D 154 in 1947. The eighth and ninth procedures are proposed as new tentatives; they are the Method of Test for Gas Checking and Draft Test of Varnish Films, and the Method of Test for Print Resistance of Varnishes. The subcommittee also prepared the revision of the Standard Specifications for Liquid Paint Driers (D 600 - 43) which is recommended for reversion to tentative. It is revising the Tentative Method of Test for Color of Transparent Liquids (Gardner Color Scale) (D 1544 - 58 T) and the Standard Methods of Testing Liquid Driers (D 564 - 47). It also has work in progress on the determination of rosin content and phenolic content of varnishes, on methods of test for polyurethane coatings, and on the color of transparent dried films.

Subcommittee X on Optical Properties (H. K. Hammond, III, chairman) is engaged in the revision of the Tentative Method of Test for Specular Gloss (D 523 - 53 T). The subcommittee has under consideration the preparation of a single recommended practice for the evaluation of color difference which is intended to coordinate and inter-relate the present five separate tentative methods of test for color difference using the following instruments: the Hunter Multipurpose Reflectometer (D 1260 - 55 T), Hunter Color Difference Meter (D 1365 - 55 T), General Electric Spectrophotometer (D 1482 - 57 T), Color Eye (D 1495 - 57 T), and Colormaster Differential Colorimeter (D 1536 - 58 T). Revisions of several of the methods listed above are also under consideration. It prepared the original drafts of the definitions of sixteen optical terms which are recommended for publication as tentative definitions. The subcommittee is preparing definitions for dominant wavelength, complementary wavelength, colorimetric purity, excitation purity, mat (matte) surface, specimen light, orange peel surface, bloom, and haze. It has begun work on a recommended practice for color matching which will include the specification of standard light sources and is completing work on a method of test for absolute hiding power. It has undertaken the measurement of tinting strength of pigments by the use of optical instruments and a study of the visual and instrumental evaluation of yellowness and whiteness.

Subcommittee XI on Resins (C. F. Pickett, chairman) prepared the two proposed Tentative Methods of Test for Phthalic Acid Isomers and Benzoic Acid in Alkyd Resins and Esters, and for Epoxy Content of Epoxy Resins. Methods for Phthalate Ester Color of High Gravity Glycerine are in preparation. The subcommittee also prepared the statement of precision which has been incorporated as an editorial addition to the Standard Method of Test for Total Nitrogen in Resins (D 1013 - 52). The subcommittee is preparing a set of definitions to describe epoxy resins and is investigating the determination of hydrolyzable and total chlorine in epoxy resins. It is determining whether the foil method for nonvolatile content, or a minor modification of it, is applicable to epoxy resins. The bubble-time method of determining viscosity is being investigated for use with resin solutions. Also under study are the determination of polystyrene in alkyd resins and a colorimetric procedure for the determination of urea in urea-formaldehyde resins; the simultaneous determination by infrared spectroscopy of urea and melamine in mixed nitrogen resins is likewise being investigated. Study of a method for the identification of fatty acids in alkyds is being continued.

Subcommittee XII on Latex and Emulsion Paints (P. T. Howard, chairman) is preparing a proposed Tentative Method of Test for Efflorescence of Interior Latex Paints. The subcommittee is continuing its development of a method of test for freeze-thaw stability and has extended this work to the related problem of evaluating the ease of re-dispersion of paints which contain gel bodies. It has prepared a group of definitions of kinds of paint failures which are characteristic of latex exterior paints, and is drafting specifications for asbestos-cement shingle "blanks" which will serve as surfaces for the exterior exposure testing of latex paints. It is continuing its studies of coalescence tests and has initiated a study of the adhesion of latex paints to chalky coatings or other coatings with weakly bound surfaces.

Subcommittee XIII on Shellac (C. C. Hartman, chairman) prepared the proposed Tentative Methods of Sampling and Testing of Shellac Varnish, and the proposed revision of the Standard

Methods of Sampling and Analysis of Shellac (D 29 - 57) which is to have the new title of Methods of Sampling and Testing Lac Resins. The subcommittee participated in cooperative tests on methods for the determination of bleach index and bleachability of seedlac, and for the determination of nonvolatile matter soluble in cold alcohol in seedlac, shellac, and bleached lac. The results of this work were transmitted through the American Standards Assn. to the Indian Secretariat of the International Committee on Lac (ISO/TC 50).

Subcommittee XIV on Statistical Application (M. P. Morse, chairman) is devising experimental designs of tests suitable for the type of cooperative work done in Committee D-1. The subcommittee is preparing recommendations for the presentation of data from cooperative tests in a manner that is meaningful and readily understood. It is also preparing definitions of statistical terms of precision, models for statements of precision, outlines of methods for calculating types of precision, and rules for the selection of data for inclusion in calculations of precision.

Subcommittee XV on Specifications for Pigments, Dry and in Oil (C. L. Crockett, chairman) prepared the proposed Tentative Specifications for Strontium Chromate Pigment, and for Basic Lead Silico-Chromate Pigment, and the revision of the Standard Methods of Sampling and Testing Aluminum Powder and Paste (D 480 - 51) with the recommendation that it be reverted to tentative. The subcommittee has under way a review of the present several specifications for iron oxide pigments D 84, D 85, D 763, D 765, D 767, and D 768. It will also consider the need for specifications for red iron oxides produced by direct precipitation without calcination.

Subcommittee XVI on Printing Inks (G. L. Erikson, acting chairman) is in the process of reorganization.

Subcommittee XVII on Flash Point (H. E. Riley, chairman) prepared the proposed revision of the Tentative Method of Test for Flash Point of Volatile Flammable Materials by Tag Open-Cup Apparatus (D 1310 - 56 T). The subcommittee is investigating further revisions of Method D 1310 and is studying techniques for testing materials with flash points below their melting points, for testing viscous materials, and for testing materials that "skin" readily.

Subcommittee XVIII on Physical Properties of Materials (J. P. McGuigan, chairman) prepared the proposed Tentative Method of Test for Moisture Vapor Permeability of Organic Coating Films. The subcommittee has drafted a method for the determination of the rheological properties of non-Newtonian materials and has begun the revision of the Tentative Method of Test for Knoop Indentation Hardness (D 1474 - 57 T). A method of test for elongation of attached coatings, using the cylindrical mandrel, has been drafted. The studies of adhesion, of the application of uniform films to non-metallic substrates, and of the measurement of settling of pigments in paints are being continued.

Subcommittee XIX on Putty, Glazing and Caulking Compounds (H. L. Kelfer, chairman) is continuing its study of accelerated weathering and has organized its fourth cooperative test in which observations on cracking, adhesion, wrinkling, bleed, and slump will be recorded at regular intervals. A fifth cooperative test has been started in the study of initial and ultimate set and surface dry. In the study of package stability and working properties, samples of compounds were allowed to age and settle without disturbance for one year and are being tested in a modified centrifuge cup. Methods for measuring stain and for measuring slump of summer grade and of winter grade caulking compounds are being investigated.

Subcommittee XXV on Lacquer (R. F. Buller, chairman) has been reorganized and has established five working groups on: wood lacquers, metal lacquers, multi-color lacquers, architectural lacquers, and film and foil. The subcommittee is completing a revision of the Standard Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 40). It has prepared a modernized definition of "lacquer" and has submitted it to Subcommittee VI on Definitions. It is engaged in the development of tests for determining the ease of re-dispersion of flatting agents, for accelerated determination of light stability and of package stability, and of a print test for lacquers applied to wood.

Subcommittee XXIX on Painting of Metals (A. J. Eickhoff, chairman) is preparing a specification to describe and classify the degrees of cleaning of ferrous surfaces for painting; suitably weathered steel panels are being cleaned by various manual and mechanical means and then photographed to provide the stereo-transparencies which will serve as pictorial reference standards. The subcommittee also has prepared drafts of specifications for the preparation of galvanized iron surfaces for painting, and for the preparation of aluminized steel surfaces for painting.

This report has been submitted to letter ballot of the committee, which consists of 376 voting members; 191 members returned their ballots, of whom 147 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

W. A. GLOGER,
Secretary.

REPORT OF COMMITTEE D-2
ON
PETROLEUM PRODUCTS AND LUBRICANTS*

This report contains the recommendations of the subordinate groups of Committee D-2 on Petroleum Products and Lubricants. The recommendations have been approved by Committee D-2 in accordance with the regulations of the Society and are presented herewith.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Gasoline (D 439 – 57 T).

Revision of Tentative Method of Test for:

Measuring the Color of Petroleum Products (ASTM Color Scale) (D 1500 – 58 T).

Withdrawal of Tentative Method for:

Calibrating Tank Car Tanks (Pressure and Non-Pressure Types) (D 1409 – 56 T).

These recommendations were accepted by the Standards Committee on September 9, 1958, and the two revised tentatives appear in the 1958 Book of ASTM Standards, Part 7.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information, or for republication as information, the following ten proposed methods:[1]

APPENDIX I. Proposed Method of Test for Smoke Density in the Flue Gases from Distillate Fuels.

APPENDIX II. Proposed Method of Test for Motor and Research Octane Numbers Permitting the Use of Small Samples.

APPENDIX III. Proposed Method of Test for Naphthalene in Aviation Turbine Fuels by Ultraviolet Spectroscopy.

APPENDIX IV. Proposed Definitions of Fluid Bulk Modulus.

APPENDIX V. Cooperative Data Showing Comparison of Results Using Apparatus Specified in Tentative Method D 1319 and "Chromanalyzer" Apparatus.

APPENDIX VI. Proposed Method of Test for Filterability of Aviation Turbine Fuels. (The proposed method will be similar to Appendix III, 1958 report of Committee D-2. New limits for filter calibration will be added.)

APPENDIX VII. Proposed Method of Test for Rust Protection of Metal Preservatives by Humidity Cabinet.

APPENDIX VIII. Proposed Method of Test for Luminosity Numbers of Aviation Turbine Fuels.

APPENDIX IX. Proposed Method of Test for Particulate Matter in Hydrocarbons.

APPENDIX X. Proposed Method of Test for Pour Stability Characteristics of Winter Grade Motor Oils.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto[2] or otherwise indicated:

Tentative Methods of Test for:

Maximum Fluidity Temperature of Residual Fuel Oils,

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The proposed methods appear in the compilation of ASTM Standards on Petroleum Products and Lubricants, October, 1959.

[2] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 7.

Thermal Stability of Aviation Turbine Fuels,
Knock Characteristics of Motor Fuels Above 100 Octane Number by the Research Method,
Specific Gravity of Light Hydrocarbons by Pressure Hydrometer,
Carbon Number Distribution of Aromatic Compounds in Naphthas by Mass Spectrometry,
Thermal Stability of Navy Special Fuel Oil (Appendix VIII, 1957 Report of Committee D-2), and
Active Sulfur in Cutting Fluids (Appendix III, 1956 Report of Committee D-2).

Tentative Specifications for:

Aviation Turbine Fuels (The proposed specifications shown in Appendix XI, 1958 Report of Committee D-2, should be revised as follows: In Section 2, change "Type C" to read "Type A-1." In Table I, change "Type C" to read "Type A-1;" under "Type B" change "Aromatics volume, max, per cent" from "25" to "20.")

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

D 96 – 58 T,[3] Tentative Method of Test for Water and Sediment by Means of Centrifuge:

Section 4 (a).—Change the first sentence to read: "Measure exactly 50 ml of industrial 90 benzene or industrial grade toluene (ASTM Specifications D 837 and D 842, respectively), saturated with water at ambient temperature into the centrifuge tube and add exactly 50 ml of the oil to be tested."

D 447 – 57 T,[3] Tentative Method of Test for Distillation of Plant Spray Oils:

Section 2.—Change "250-ml flask" to "125-ml flask."

Section 3.—Change to read as follows:

3. All items listed in Paragraphs (*a*) to (*g*) shall conform to the ASTM Specifications for Distillation Equipment (ASTM Designation: E 133); all sections and figure numbers refer to these specifications, rather than to the text of this method:

(*a*) *Distilling Flask.*—Flask B (125 ml), as shown in Fig. 3.

(*b*) *Condenser and Cooling Bath.*—Section 4; Figs. 1 and 2.

(*c*) *Shield.*—Section 5; Figs. 1 and 2.

(*d*) *Heater.*—Section 6; Figs. 1 and 2.

(*e*) *Flask Support.*—Section 7; Table II, Board C (2.0-in. hole). An additional asbestos board, which will completely cover the top of the shield, is split and recessed to fit the neck of the flask.

(*f*) *Graduated Cylinder.*—Section 8; Graduate B, 100 ml, as shown in Fig. 4.

(*g*) *Thermometer.*—Section 9; ASTM Thermometer 8 F.

NOTE 1.—Thermometers heated to high temperatures, in the range required for spray oil distillations, sometimes develop stresses which may affect the accuracy of calibration. It is recommended that, when thermometers vary from the standard thermometer when checked at any convenient temperature, the thermometers be allowed to rest at room temperature for at least 24 hr to relieve stresses.

(*h*) *Water Bath.*—A suitable water bath maintained at boiling temperature, in which the cylinder can be immersed to the top graduation mark.

Notes 1 and 3.—Delete these notes, renumbering remaining notes accordingly.

Section 4 (c).—Change "2¾-in. hole" to "2.0-in. hole."

Section 5 (a).—In the equation change "Cf" to "C_f."

Appendix.—Delete this appendix; it will be replaced by Specifications E 133.

D 613 – 58 T,[4] Tentative Method of Test for Ignition Quality of Diesel Fuels by the Cetane Method:

The following change is recommended in Appendix I of Method D 613 which appears in the 1959 ASTM Manual for Rating Diesel Fuels by the Cetane Method:

Section 112.—Add the following sentences after the first sentence (of the new Cetane method manual):

---

[3] 1958 Book of ASTM Standards, Part 7.

[4] ASTM Manual for Rating Diesel Fuels by the Cetane Method, 1959.

As this method is quite sensitive to inlet air temperature, line voltage fluctuations or air drafts in the room make an automatic temperature controller desirable. The Waukesha temperature controller, available from Waukesha Motor Co., Waukesha, Wis., is approved for this purpose. The small thermistor pickup for this instrument is screwed into the side of the air inlet elbow, with the control dials and saturable reactor mounted in the console panel.

D 614 - 58 T,[5] Tentative Method of Test for Knock Characteristics of Aviation Fuels by the Aviation Method:

The following changes are recommended in Appendices I and II of Method D 614 which appear in the 1958 ASTM Manual for Rating Aviation Fuels by Supercharge and Aviation Methods:

Section 115.—Delete the last three sentences and substitute the following:

> The Malraison mixture thermostat and the Waukesha temperature controller are approved for this purpose. The Malraison unit consists of a bimetallic thermostatic element incorporated in a ⅜-in. spacer between the intake manifold and the intake port, and an electronic relay unit mounted in the console panel. The Waukesha controller uses a small thermistor pickup which screws into a boss on the manifold with a variable control dial and a saturable reactor mounted in the console panel. The Malraison thermostat is available from P. J. Malraison, Mt. Vernon, N. Y., and the Waukesha temperature controller from the Waukesha Motor Co., Waukesha, Wis.

Section 202 (a).—In the first sentence of the second paragraph change "octane range above 60" to read "performance number range between 77 and 120." In the third sentence change ". . . . vary respectively from 0 to 75 and *vice versa*" to "are varied respectively to cover the practical range of fuel testing."

Table XIV.—Delete and replace with a new Table XIV as shown in the accompanying Table I.

Section 202 (b).—Delete everything but the first sentence.

TABLE I.—ASTM AVIATION RATINGS OF STANDARDIZATION FUEL BLENDS OF BENZENE, *ISO*OCTANE, *n*-HEPTANE, AND TETRAETHYLLEAD.

(New Table XIV, Method D 614)

Aviation Performance Number	Composition of Blends by Volume			
	Iso-octane, per cent	*n*-Heptane, per cent	Benzene, per cent	Tetraethyllead, ml per U. S. gal
77.0	50	25	25	4.0
79.2	51	24	25	4.0
81.4	52	23	25	4.0
83.6	53	22	25	4.0
85.8	54	21	25	4.0
87.9	55	20	25	4.0
90.1	56	19	25	4.0
92.3	57	18	25	4.0
94.5	58	17	25	4.0
96.6	59	16	25	4.0
98.8	60	15	25	4.0
101.0	61	14	25	4.0
103.2	62	13	25	4.0
105.4	63	12	25	4.0
107.5	64	11	25	4.0
109.7	65	10	25	4.0
111.9	66	9	25	4.0
114.1	67	8	25	4.0
116.2	68	7	25	4.0
118.4	69	6	25	4.0
120.6	70	5	25	4.0

D 874 - 57 T,[3] Tentative Method of Test for Sulfated Residue from New Lubricating Oils:

Title.—Change "Residue" to "Ash."

Sections 1 (a) and (b), 2, 6 (e), 7, 8 (a) and (b).—Change "residue" to "ash."

New Section.—Insert a new Section 3 entitled "Definition" to read as follows, renumbering subsequent sections accordingly:

> 3. *Sulfated Ash.*—The ash that remains after the sample has been carbonized, and the residue subsequently heated with sulfuric acid to constant weight.

---

[5] ASTM Manual for Rating Aviation Fuels by Supercharge and Aviation Methods, 1958.

D 909 – 58 T,[5] Knock Characteristics of Aviation Fuels by the Supercharge Method:

The following changes are recommended in Appendices I, II, and III of Method D 909 which appear in the 1958 ASTM Manual for Rating Aviation Fuels by Supercharge and Aviation Methods:

Section 111 (d).—Delete and replace with the following:

> (*d*) The maximum permissible back pressure at the surge tank is 10 in. of water, unless low barometric pressure prevents meeting the requirements of Sections 8(*a*) and (*b*). In this case, the standard exhaust surge tank back pressure may be increased to the difference between the prevailing barometer and 29.92 in. of mercury.

Section 202 (a).—In the third sentence of the first paragraph, change "above 85 octane number" to "above 87 performance number." In the fifth sentence, change ". . . . vary respectively from 0 to 75 and *vice versa*" to "are varied respectively to cover the practical range of fuel testing." Delete the last sentence and replace with "The calibration values are shown in Table V."

Table V.—Delete and replace with a new Table V as shown in the accompanying Table II.

Figs. 8 and 9.—Delete and renumber remaining figures accordingly.

Section 202 (b).—Delete everything but the first sentence.

Section 312 (a).—Add a sentence after the fourth sentence to read: "In areas where this requirement cannot be met because of low barometric pressure, refer to Section 111 (*d*) for corrective information."

Section 312 (b).—After the second sentence add a sentence to read: "In areas where this requirement cannot be met because of low barometric pressure, refer to Section 111 (*d*) for corrective information."

Section 317.—Change Item (*13*) to read: "Resonance in exhaust system." Insert the following as Item (*14*), renumbering subsequent items accordingly: "Excessive back pressure or, in the case of low barometric pressures, particularly in altitude laboratories, insufficient back pressure, see Section 111 (*d*)."

TABLE II.—ASTM SUPERCHARGE (RICH) RATINGS OF STANDARDIZATION FUEL BLENDS OF BENZENE, *ISO*OCTANE, *n*-HEPTANE AND TETRAETHYLLEAD.

(New Table V, Method D 909)

Supercharge Performance Number	Composition of Blends by Volume			
	Iso-octane, per cent	*n*-Heptane, per cent	Benzene, per cent	Tetraethyllead, ml per U. S. gal
87.0	50	25	25	4.0
90.0	51	24	25	4.0
93.1	52	23	25	4.0
96.2	53	22	25	4.0
99.3	54	21	25	4.0
102.4	55	20	25	4.0
105.5	56	19	25	4.0
108.6	57	18	25	4.0
111.7	58	17	25	4.0
114.8	59	16	25	4.0
117.9	60	15	25	4.0
121.0	61	14	25	4.0
124.0	62	13	25	4.0
127.1	63	12	25	4.0
130.2	64	11	25	4.0
133.3	65	10	25	4.0
136.4	66	9	25	4.0
139.5	67	8	25	4.0
142.6	68	7	25	4.0
145.7	69	6	25	4.0
148.8	70	5	25	4.0

D 975 – 53 T,[3] Tentative Classification of Diesel Fuel Oils:

Table I.—Revise as follows: Remove the "End Point" column under "Distillation Temperatures, deg Fahr," and replace with "90 per cent Distillation Temperatures, deg Fahr, min and max." Add for grade No. 1-D, a maximum 90 per cent distilled of 550 F, and a maximum kinematic viscosity at 100 F

of 2.5 cs (34.4 sec). Change footnote designation for "Cetane Number" from "*c*" to "*d*," and for cetane numbers for grades No. 1-D, No. 2-D, and No. 4-D from "*d*" to "*c*." Add for grade No. 2-D a minimum 90 per cent distilled of 540 F.[e] Change minimum kinematic viscosity at 100 F for grade No. 2-D from "1.8 cs (32.0 sec.)" to "2.0 cs[e] (32.6 sec).[e]"

Reletter Footnote *d* as "*c*." Reletter Footnote *c* as "*d*." Add a new Footnote *e* to read: "[e]When pour point less than 0 F is specified, the minimum viscosity shall be 1.8 cs (32.0 sec) and the minimum 90 per cent point shall be waived."

D 1022 – 49 T,[3] Tentative Method of Test for Peroxides in Butadiene (Ferrous-Titanous Method):

New Note.—In Section 4 (*b*) add a new Note 3 after the first sentence to read as follows, renumbering the present Note 3 and reference to it accordingly: "NOTE 3.—Samples from the *original source* must be taken in a stainless steel container."

Section 6.—Change to read as follows:

6. The following data should be used for judging acceptability of results (95 per cent confidence):

Repeatability.—Duplicate results obtained by the same operator should not be considered suspect unless they differ by more than 2 ppm.

Reproducibility.—Results submitted by each of two laboratories should not be considered suspect unless they differ by more than 4 ppm.

D 1158 – 57 T,[3] Tentative Method of Test for Bromine Number of Petroleum Distillates (Color-Indicator Method):

Note.—Insert a note across the title page to read: "The committee responsible for the development of this method urges that its use be discontinued and that ASTM Method D 1159, Test for Bromine Number of Petroleum Distillates (Electrometric Method) be used in its place. The committee will consider recommending the withdrawal of Method D 1158 as an ASTM tentative in 1960."

D 1159 – 57 T,[3] Tentative Method of Test for Bromine Number of Petroleum Distillates (Electrometric Method):

Section 5 (*a*).—Change to read as follows:

(*a*) *Titration Solvent.*—Prepare 1 liter of titration solvent by mixing the following volumes of materials: 714 ml of glacial acetic acid, 134 ml of carbon tetrachloride, 134 ml of methanol, and 18 ml of sulfuric acid (1:5).

Section 6.—Change the third sentence to read as follows:

If the reagents and techniques are correct, values within the following ranges should be obtained:

Standard	Bromine Number
Cyclohexene, purified (Notes 1 and 2)	187 to 199 (Note 3)
Cyclohexene, 10 per cent solution	18 to 20
Diisobutene, purified (Notes 1 and 2)	136 to 144 (Note 3)
Diisobutene, 10 per cent solution	13 to 15

Note 1.—In the first sentence of the second paragraph change the phrase within parentheses to read: "(100 to 200 mesh, manufactured to insure minimum olefin polymerization).[4]" In the third paragraph change the volumes "30 ml," "15 ml," and "10 ml" to read "30 ml," "10 ml," and "10 ml," respectively.

Footnote.—Add the following footnote 4:

[4] Available from Davison Chemical Corp., Baltimore 3, Md., by specifying Code 923.

Section 9.—Change to read as follows:

9. The following criteria should be used for judging the acceptability of results:

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than the amounts shown in Fig. 1.

(*b*) ***Reproducibility.***—Results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the amounts shown in Fig. 2.

Appendix II.—Add a new Appendix II as appended hereto[6] showing the behavior of pure hydrocarbons when using the proposed new titration solvent. Number the present Appendix as I.

D 1220 – 58 T,[3] Tentative Methods for Calibrating Upright Tanks:

Section 51 (*d*).—Add the following sentence at the end of Note 11: "The corrected strapped circumference is then stressed to a "ring full" basis by expanding the unstressed circumference for each ring by the height of liquid above the circumferential elevation necessary to fill each ring."

Replace Eqs 5, 6, and 7 with the following revised equations:

Volume correction per increment, Δv:

$$\Delta v \text{ (first, bottom, ring)} = 0 \quad \text{(5)}$$

$$\Delta v \text{ (second ring)} = \frac{\pi W d^3}{4E}\left(\frac{h_1}{t_1}\right) \quad \text{(6)}$$

$$\Delta v \text{ (third ring)} = \frac{\pi W d^3}{4E}\left(\frac{h_1}{t_1} + \frac{h_2}{t_2}\right) \quad \text{(7)}$$

$$= \text{etc.}$$

where:

Δv = additional tank volume resulting from tank shell expansion due to increased head of an increment one unit deep above the ring,

W = weight of liquid per unit volume,

d = nominal tank diameter,

E = modulus of elasticity of metal in tank shell,

h_1, h_2, etc. = height of shell rings, and

t_1, t_2, etc. = thickness of shell rings.

Note 12.—Change to read as follows:

NOTE 12.—All units must be consistent. For example, in the English system, Δv may be in cubic inches; W in pounds per cubic inch; d, h, and t in inches, and E in pounds per square inch. The increment corresponding to Δv is 1 in. If the gage table is made in ¼-in. increments, Δv must be divided by 4. In the metric system, Δv may be in cubic centimeters; W in grams per cubic centimeter; d, h, and t in centimeters; and E in grams per square centimeter. The increment corresponding to Δv is 1 cm.

Appendix I.—Replace the "Example of Calculations for Upright Cylindrical Steel Tank, Above Ground" with the similar proposed example shown on pp. 1145 through 1151 of the 1958 Book of ASTM Standards, Part 7, renumbering Table XII and references thereto as Table VI. Retain Tables VII, VIII, IX, X, XI, and Appendices II and III.

D 1266 – 57 T,[3] Tentative Method of Test for Sulfur in Petroleum Products Including Liquefied Petroleum Gas by Lamp Combustion:

Section 1 (*a*).—Change to read as follows:

1. (*a*) This method describes procedures for the determination of total sulfur in liquid petroleum products in concentrations above 0.002 per cent by weight and combined sulfur in liquid petroleum gas or in light hydrocarbon mixtures boiling in this range (Note 1). A special sulfate analysis procedure is described in Appendix I that permits the determination of sulfur in concentrations as low as 1 ppm.

NOTE 1.—The comparable lamp method for the determination of sulfur in fuel gases is described in ASTM Method D 1072. For the determination of sulfur in heavier petroleum products that cannot be burned in a lamp, see the bomb method (ASTM D 129), the quartz tube method (ASTM D 1551), or the high-temperature combustion method (ASTM D 1552).

Footnote 2.—Change to read as follows:

[2] Latest revision accepted by the Society at the Annual Meeting in June, 1959. In 1959, the method was revised by the addition of Appendix I permitting the determination of trace quantities of sulfur by turbidimetric analysis.

Section 2.—In Paragraphs (*a*) and (*b*) change "(see Appendix I)" to read "(see

---

[6] The new Appendix is not included in this report but appears in the revised Method of Test, see 1959 Supplement to Book of ASTM Standards, Part 7.

Appendix II)." Add a new Paragraph (*c*) to read as follows:

(*c*) For sulfur contents below 0.002 per cent by weight, it is necessary to determine the sulfate content in the absorber solution turbidimetrically as barium sulfate (see Appendix I).

Section 3.—In Paragraph (*a*) change "Appendix II" to "Appendix III."

Change Paragraph (*b*) to read as follows:

(*b*) *Cotton Wicking.*—Clean, unused, uniform, twisted white cotton yarn of good quality.[4] For the burner to burn aromatic samples use long staple, fine spun, commercial *fine* grade.

In Paragraph (*c*) change "Appendix II" to "Appendix III" and "Appendix I" to "Appendix II."

Section 8.—Change "(see Appendix I)" to "(see also Appendix II)."

Section 10 (*e*).—Change "(Appendix II, Section A-10)" to "(Appendix III, Section A-19)."

Note 17.—Delete reference to NGAA publication No. 2140-55, and replace with ASTM Method D 1657, Test for Specific Gravity of Light Hydrocarbons by Pressure Hydrometer.[6]

Appendix I.—Add a new Appendix I, "Trace Quantities of Sulfur," as appended hereto.[6] Renumber the present Appendix I as Appendix II and make the following changes therein: Renumber Sections A-1 through A-8 as A-10 through A-17. In the present Section A-6 change "Note 19" to "Note 25" and in the present Section A-7 change "Note 20" to "Note 26." Renumber the present Appendix II as Appendix III and make the following changes therein: Renumber Sections A-9 through A-15 as A-18 through A-24. In the present Section A-15 change "Note 21" to "Note 27."

D 1318 – 54 T,[3] Tentative Methods of Test for Sodium in Residual Fuel Oils by Flame Photometer: A complete revision of the method is proposed as appended hereto,[7] in order to make it more applicable to current photometers. The precision section has been rewritten.

D 1322 – 58 T,[3] Tentative Method of Test for Smoke Point of Jet Fuels:

Section 2.—Delete the last sentence.

Section 5.—Add a line to the tabulation to list a fuel of 0 per cent toluene, 100 per cent *iso*octane, and smoke point of 42.8 at 760 mm. Also change the smoke point of 5 per cent toluene blend from "35.0" to "35.4."

Figures 3 and 4.—Delete these figures.

Section 7 (*b*).—Change to read as follows:

(*b*) The required reference fuel blends should be run each day by each operator. A test on a given reference fuel blend need not be repeated in any given day unless a change in the apparatus or operator is made, or a change of more than 5 mm occurs in the barometric pressure reading.

Section 8 (*e*).—Delete the last sentence.

Section 9 (*a*).—Change the second sentence to read: "Correct this average by use of the correction factor obtained in Section 7."

Section 10.—Change to read as follows:

10. The following data should be used for judging the acceptability of results and two results should not differ from each other by more than the following amounts (95 per cent probability):

Smoke Point	Repeatability, Same Operator and Apparatus	Reproducibility, Different Operators and Apparatus
Up to 20..........	1	2
20 to 30..........	1	3
30 to 40..........	1	4

D 1548 – 58 T,[3] Tentative Method of Test for Vanadium in Navy Special Fuel Oil:

Note 1.—Delete this Note, renumbering subsequent notes and references thereto accordingly.

---

[7] The revised Methods of Test appear in the 1959 Supplement to Book of ASTM Standards, Part 7.

Section 5.—Delete Paragraphs (*b*) and (*c*) and reletter the remaining paragraphs accordingly.

Reletter Paragraph (*j*) as (*h*) and change to read as follows:

(*h*) Place 0.4463 g of vanadium pentoxide (V_2O_5) (Note 2) in a 400-ml beaker and treat it as described in Sections 8(*f*) and (*g*), except add 4 ml of H_2SO_4 rather than the 2 ml specified in Section 8(*f*). Wash carefully with distilled water into a volumetric flask and dilute to 500 ml.

Note 3.—Renumber as Note 2 and add a sentence to read: "If the V_2O_5 is less than 99 per cent pure, weigh out a correspondingly larger amount than the 0.4463 g specified above."

Section 8.—Change Paragraph (*f*) to read as follows:

(*f*) *Color Development.*—To the cool beaker containing the inorganic sulfated ash add the following acids in the order listed: 2 ml of HNO_3 (sp gr 1.42) and 2 ml of H_2SO_4 (sp gr 1.84). Heat carefully on a hot plate until most of the nitric acid is evaporated and white fumes just begin to appear. At this point remove from the hot plate (Note 6).

Note 7.—Delete and replace with the following new Note 6:

NOTE 6.—Heating at the temperature of fuming sulfuric acid in the absence of nitric acid may cause low results due to reduction of pentavalent vanadium.

Change Paragraph (*g*) to read as follows:

(*g*) Wash the sides of the beaker cautiously with about 15 ml of water, heat to boiling, and cool. Neutralize by adding NaOH solution dropwise until the pH is between 6 and 10 as shown by placing a fraction of a drop (Note 7) on wide range pH paper.[7] Add a few drops of H_2SO_4 (1:1) until the pH of the solution is between 3 and 5.

Note 7.—Add a new Note 7 to read as follows:

NOTE 7.—Very small amounts of solution should be withdrawn in the pH test measurements to insure that significant amounts of vanadium are not lost. This is conveniently done by wetting the pH test paper with a very small amount of solution on the end of a stirring rod which has been drawn out to about 1 mm in diameter.

New Footnote.—Add the following new footnote 7: "[7]Wide range Hydrion paper, A or B has been found satisfactory."

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends the revision and reversion to tentative of the Standard Method of Test for Ash Content of Petroleum Oils (**D 482 – 46**)[3] as appended hereto.[7]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Test for Ramsbottom Carbon Residue of Petroleum Products (**D 524 – 58 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard, revised as appended hereto.[7]

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

D 86 – 56,[3] Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products: Replace the present text of the method with the text shown in Appendix XIII, Test for Distillation of Petroleum Products, to the 1958 Report of Committee D-2.[7]

D 128 – 57,[3] Standard Methods of Analysis of Lubricating Grease: Include as an Appendix to the standard the Proposed Optional Method for Analysis of Lubricating Greases appended as information in the 1958 Report of Committee D-2:[7]

D 158 – 54,[3] Standard Method of Test for Distillation of Gas Oil and Similar Distillate Fuel Oils:

New Note.—Insert a note under the title to read: "The committee responsible for the development of this method calls attention to the fact that products covered by its scope can be tested by ASTM Method D 86 – 59. The committee will consider recommending the withdrawal of Method D 158 as an ASTM standard in 1960."

D 357 – 58,[3] Standard Method of Test for Knock Characteristics of Motor Fuels by the Motor Method:

Section 5.—Delete footnotes 5 and 6 which refer to fuels "C" and "X," and all references to them.

Section 6 (j).—Change to read as follows:

(j) Intake Air Humidity, 25 to 50 grains of water per pound of dry air (as described in Section 114 of Appendix I). When intake air at this humidity and atmospheric pressure is available through the operation of natural factors or air conditioning, it may be used without further conditioning, its humidity being determined by use of the type of sling psychrometer described in ASTM Method D 337, for Determination of Relative Humidity.[8]

Section 10 (a).—Revise to read as follows:

10. (*a*) Engine conditions are not correct unless a blend of 74 per cent toluene and 26 per cent *n*-heptane by volume rates 81.1 ± 0.3 octane number. If this match cannot be obtained under standard conditions, check the mechanical condition of the engine. Other standardization fuels (Section 203 of Appendix II) may be used for checking both engine and test conditions at other octane number levels. Their rating should fall within plus or minus two standard deviations of the value given in the appropriate certified calibration table. Standard deviation in this sense is that appropriate for the laboratory performing a test at the octane number level considered.

Section 14.—Change to read as follows:

14. ***Reproducibility.***—(*a*) Reproducibility[1] is a quantitative measure of the variability associated with operators working in two different laboratories. It is defined as the greatest difference between a single test result obtained in one laboratory on a given fuel and a single result obtained in another laboratory on the same fuel that need not be considered suspect (significantly different) at a given probability level, usually 95 per cent. Within the meaning of this method, differences smaller than this reproducibility limit are considered identical, statistically.[3] Extensive data from consumer and producer laboratories over a number of years for many samples of conventional motor gasolines have shown that the variability[2] (standard deviation) of fuel ratings between laboratories varies with octane number level. This variation is shown in the accompanying Fig. 2(*a*), where standard deviation is plotted against octane number level. The curve for this figure is based on data obtained by laboratories (15 to 30) participating in the National Exchange Group cooperative test program for the period January, 1947, through May, 1958 inclusive.

(*b*) The allowable difference in ratings between two laboratories while testing portions from the same sample shall be determined as follows:

(*1*) Average the test results obtained by the two laboratories.

(*2*) Read from the curve of Fig. 2(*a*) the standard deviation for the octane level calculated as the average in Item (*1*) above.

(*3*) Multiply the standard deviation read from the curve, in Item (*2*) above, by 2.772.[3] The product of this multiplication is the greatest difference between single results obtained by two laboratories that can be tolerated without being suspect at the 95 per cent[3] probability level.

(*c*) Reproducibility data given herein apply only to conventional motor gasolines when careful attention is given to the details of the test procedure and engine conditions. Similar data for fuels which differ materially from finished motor gasolines is likely to indicate poorer reproducibility (higher standard deviation) than shown here.

---

[8] 1958 Book of ASTM Standards, Part 10.

New Footnotes.—Add footnotes *1*, *2*, and *3* as follows:

[1] See Proposed Recommended Practices for Applying Precision Data, given in ASTM Methods of Test for Petroleum Products and Lubricants, Directory of Committee D-2, January, 1958.

[2] For a more complete ASTM publication on the presentation of data in terms of limits of uncertainty about an observed average, see "ASTM Manual on Quality Control of Materials," *ASTM STP 15-C*, January, 1951.

[3] If 90 per cent probability level is desired, the factor 2.772 (95 per cent confidence) becomes 2.327 and, similarly, for 99 per cent probability the factor is 3.645. If the test result of a third or referee laboratory is to be compared with those of two laboratories (which failed to agree within the acceptable limits for reproducibility), then for 90 per cent probability the factor is 2.903; for 95 per cent probability, 3.315; and for 99 per cent probability the factor becomes 4.123. For this latter purpose, the average for all three laboratory ratings is used to read the standard deviation from the curve for calculating the allowable difference in rating between the three laboratories.

New Figure.—Add a new Fig. 2(*a*) as shown in the accompanying Fig. 1.[9]

New Section.—Add a new Section 15 to read as follows:

15. ***Repeatability.***—Repeatability is a quantitative measure of the variability associated with a single operator in a given laboratory, generally with the same apparatus and within a small interval of time. It is defined as the greatest difference between two single and separate results on the same fuel that can be considered acceptable at a given probability level, usually 95 per cent. Repeatability may not be read from Fig. 2(*a*). Each laboratory must determine its own within-laboratory standard deviation on the fuel types normally tested to obtain a measure of its test repeatability. The standard deviation when multiplied by the appropriate factors given in Section 14 will yield ***repeatability*** figures for sets of two or three ratings. In general, repeatability figures, in terms of standard deviations, will be smaller than the corresponding reproducibility figures. In the unusual case where this is not true and it is desired to reduce the standard deviation for repeatability to that indicated for reproducibility by ***replicate*** testing, then the number of tests required to yield ***equal*** variability may be determined by dividing the square of the repeatability by the square of the reproducibility, both at the same confidence level. Usually, this division will not result in a whole number. In this latter case, the number of tests required shall be the next larger whole number. This ***reduced repeatability*** figure applies only to the average of the number of tests indicated by the above calculation at the rating level employed. If a repeatability figure better than that for reproducibility is required, then the ***desired*** standard deviation figure is substituted for that shown for reproducibility before proceeding with the calculation as noted above to determine the number of replicate ratings whose average will have the desired degree of precision.

In addition, the following changes are recommended in Appendices I, II, and IV of Method D 357 which appear in the 1956 ASTM Manual for Rating Motor Fuels by Motor and Research Methods:

Section 101 (*d*).—In Table VII revise information on piston rings as follows:

Compression Rings:		
Number Required		4
Type	Top	straight-sided, chromium plated, or unplated
	Other Three	straight-sided, unplated
Oil Control Rings:		
Number Required		1
Type		85

Section 106.—Change the wording under "Rings" to read as follows:

Rings.—Two sets of straight-sided piston rings are approved. The preferred set consists of a chromium-plated top compression ring, Waukesha Part No. 106222-B, three unplated compression rings, Waukesha Part No. 106222-A, and a ventilated oil control ring, Waukesha Part No. 23505. With the other set, the unplated ring, Waukesha Part No. 106222-A, is used in the top ring groove. The four other rings are the same for both sets.

Section 113.—Insert the following as the last paragraph: "An inlet air thermostat may be used to improve temperature control, especially on the Research Method. Approved instruments are the

---

[9] This figure has been incorporated in the revised method which appears in the 1959 Supplement to Book of ASTM Standards, Part 7.

Malraison thermostat made by P. J. Malraison, Mt. Vernon, N. Y., and the Waukesha temperature controller, made by the Waukesha Motor Co., Waukesha, Wis."

Section 115.—Replace the last three sentences with the following:

The Malraison mixture thermostat and the Waukesha temperature controller are approved for this purpose. The Malraison unit consists of a bimetallic thermostatic element incorporated in a ⅜-in. spacer between the intake manifold and the intake port, and an electronic relay unit mounted in the console panel. The Waukesha controller uses a small thermistor pickup which screws into a boss on the manifold with a variable control dial and a saturable reactor mounted in the console panel.

TABLE III.—ASTM OCTANE NUMBER CONVERSION FOR STANDARDIZATION FUEL BLENDS OF TOLUENE AND *n*-HEPTANE; TOLUENE, *ISO*OCTANE, AND *n*-HEPTANE; AND TOLUENE AND *ISO*OCTANE.

Octane Number, Research Method ASTM D 908	Volume, per cent			Octane Number Motor Method ASTM D 357
	Reference Fuel Grade Toluene	ASTM Grade		
		*Iso*octane	*n*-Heptane	
65.2	50	0	50	57.8
85.0	66	0	34	74.4
93.4	74	0	26	81.1
99.6	74	10	16	88.5
103.3	74	15	11	92.5
108.0	74	20	6	96.8
113.7	74	26	0	100.8

Section 202.—Delete this section, renumbering subsequent sections accordingly.

Section 203.—In Paragraph (*a*) put a period after "methods" in the first sentence and delete the rest of the sentence. Delete footnote *2* and the reference to it.

In Paragraph (*b*) delete the last sentence.

In Paragraph (*c*) change the second sentence to read: "Research Division I has provided Additional Standardization Fuels containing various proportions of some or all of the reference materials listed in Section 214(*a*) and (*e*)."

Revise Item (*1*) to read as follows:

(*1*) A series of blends by volume of toluene and *n*-heptane; or toluene, *iso*octane and *n*-heptane; or toluene and *iso*octane have been calibrated by the Division to supply this need. Table VIIA lists verified calibration data and Section 215 identifies the reference fuel suppliers.

Table VIIA.—Add a new Table VIIA as shown in the accompanying Table III.

Section 204.—In Paragraph (*a*) delete the phrase "except for toluene or X fuel" from the last sentence.

In Paragraph (*b*) delete sentences three and four and the word "However" in sentence five. In sentence six, delete the words "reference and."

Section 212 (*b*).—Add a period after "fuels" and delete the rest of the sentence. Delete the second sentence.

Section 215.—Delete "Secondary Reference Fuel C" and "Standardization Fuels X and XH" from the list of reference materials.

Section 416 (*d*).—Change to read as follows:

(*d*) *Checking Piston Rings.*—New or used piston rings should conform to the following measurements before installing or reinstalling on the piston:

Cast Iron Pistons	Measurements, in.
Gap Clearance:	
New Compression Rings	0.007 to 0.017
New Oil Rings	0.010 to 0.018
Gap Clearance, Replacement Limits:	
Compression Rings:	
Chromium Plated	0.030[a]
Unplated	0.030[a]
Oil Rings	0.030[a]
Side Clearance:	
Top Groove	0.001 to 0.003
All Others	0.001 to 0.0025

[a] Or when the surface shows signs of scuffing or scoring.

Compression and oil control rings should be changed when their gap clearance exceeds 0.030 in. To measure gap clearance, the ring should be placed in the cylinder barrel and the gap measured witb feeler gages. To insure correct alignment, the ring should be pushed into the cylinder with the piston to a depth of 1½ in. The piston ring grooves also wear. If the side clearance with a new piston ring is too great, a new piston should be installed.

D 473 - 48,[3] Standard Method of Test for Sediment in Fuel Oil by Extraction:

Section 1.—Change to read: "1. This is a method of determining the sediment in fuel oil by extraction with benzene."

New Section.—Add a new Section 2, entitled "Outline of Method," to read as follows, renumbering subsequent sections accordingly:

2. *Outline of Method.*—A 10-g sample, in an alundum thimble, is extracted with hot benzene until the residue reaches constant weight. The weight of residue, calculated to per cent, is reported as "sediment by extraction."

Section 2.—In Paragraph (*a*) put a period after "capacity" and delete the rest of the sentence.

In Paragraph (*c*) change the second sentence to read: "The thimble shall be suspended from the condenser coil by means of a corrosion-resistant wire, made of platinum, stainless steel, Nichrome, or similar material so that it hangs . . . coil."

In Paragraph (*d*) change "benzol" to "benzene."

Section 3.—Add a new first sentence to read: "Before using a new alundum thimble, rub the outside surface with fine sandpaper and remove all loosened material with a stiff brush. Give the thimble . . . 1 hr. Then dry. . . ." Also change the solvent "industrial 90 benzene (ASTM Specifications D 837)" to "nitration-grade benzene (ASTM Specifications D 835[10])," and the word "benzol" to "benzene."

Section 4.—Change the title to "Precision" and revise to read as follows:

4. ***Precision.***—Two results on the same sample should be considered suspect if they differ by more than the following amounts (95 per cent probability):

Range of sediment, per cent	0.0 to 0.4
Repeatability (one operator and apparatus)	$0.010 + 0.175S$
Reproducibility (different laboratories)	$0.024 + 0.38S$

where S = average per cent sediment by extraction.

Figure 1.—Change "Platinum Wire" to read "Corrosion-Resistant Wire."

D 810 - 48,[3] Standard Methods of Test for Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils:

Section 1.—Change title to "Scope and Applications."

Note 1.—Change title of D 874 to "Test for Sulfated Ash." Delete "sulfated" from the first sentence.

New Section.—Add a new Section 3 entitled "Definition" to read as follows, renumbering subsequent sections accordingly:

3. *Sulfated Residue.*—The residue that remains after the sample has been digested with sulfuric and nitric acids and subsequently heated to constant weight.

---

[10] 1958 Book of ASTM Standards, Part 8.

D 908 – 58,[4] Standard Method of Test for Knock Characteristics of Motor Fuels by the Research Method: The revisions proposed are identical to those recommended above for Motor Method D 357, except for the sections listed below, which should appear in the Research Method as indicated:

Section 6 (j).—Change to read as follows:

(*j*) *Intake Air Humidity*, 25 to 50 grains of water per pound of dry air (as described in Section 114 of Appendix I). When intake air at this humidity and atmospheric pressure is available through the operation of natural factors or air conditioning, it may be used without further conditioning, its humidity being determined by use of the type of sling psychrometer described in ASTM Method D 337, for determination of relative humidity.[8]

Section 10 (a).—Change to read as follows:

(*a*) Engine conditions are not correct unless a blend of 74 per cent toluene and 26 per cent *n*-heptane by volume rates 93.4 ± 0.3 octane number. If this match cannot be obtained under standard conditions, check the mechanical conditions of the engine. Other standardization fuels (Section 203 of Appendix II) may be used for checking both engine and test conditions at other octane number levels. Their rating should fall within plus or minus two standard deviations of the value given in the appropriate certified calibration table. Standard deviation in this sense is that appropriate for the laboratory performing a test at the octane number level considered.

Section 14.—Revise to read as follows:

14. *Reproducibility.*—(*a*) Reproducibility[1] is a quantitative measure of the variability associated with operators working in two different laboratories. It is defined as the greatest difference between a single test result obtained in one laboratory on a given fuel and a single result obtained in another laboratory on the same fuel that need not be considered suspect (significantly different) at a given probability level, usually 95 per cent. Within the meaning of this method, differences smaller than this reproducibility limit are considered identical, statistically.[3] Extensive data from consumer and producer laboratories over a number of years for many samples of conventional motor gasolines have shown that the variability[2] (standard deviation) of fuel ratings between laboratories varies with octane number level. This variation is shown in the accompanying Fig. 3(*a*) where standard deviation is plotted against octane number level. The curve for this figure is based on data obtained by laboratories (15 to 30) participating in the National Exchange Group cooperative test program for the period January, 1947, through May 1, 1958, inclusive.

(*b*) The allowable difference in ratings between two laboratories while testing portions from the same sample shall be determined as follows:

(*1*) Average the test results obtained by the two laboratories.

(*2*) Read from the curve of Fig. 3(*a*) the standard deviation for the octane level calculated as the average in Item (*1*) above.

(*3*) Multiply the standard deviation read from the curve, in Item (*2*) above, by 2.772.[3] The product of this multiplication is the greatest difference between single results obtained by two laboratories that can be tolerated without being suspect at the 95 per cent[3] probability level.

(*c*) Reproducibility data given herein apply only to conventional motor gasolines when careful attention is given to the details of the test procedure and engine conditions. Similar data for fuels which differ materially from finished motor gasolines is likely to indicate poorer reproducibility (higher standard deviation) than shown here.

New Footnotes.—Add footnotes *1*, *2*, and *3* as indicated above for new footnotes *1*, *2*, and *3* in Methods D 357.

New Figure.—Add new Fig. 3(*a*) as shown in the accompanying Fig. 2.[9]

New Section.—Add a new Section 15 to read as follows:

15. *Repeatability.*—Repeatability is a quantitative measure of the variability associated with a single operator in a given laboratory, generally with the same apparatus and within a small interval of time. It is defined as the greatest difference between two single and separate results on the same fuel that can be considered acceptable at a given probability level, usually 95 per cent. Repeatability may not be read from Fig. 3(*a*). Each laboratory must determine its own within-laboratory standard deviation on the fuel types normally tested to obtain a measure of its test repeatability. The standard deviation when multiplied by the appropriate factors given in Section 14 will yield ***repeatability*** figures for sets of two or three ratings. In general, repeatability figures, in terms of standard deviations, will be smaller than the corresponding reproducibility figures. In the unusual case where this is

not true and it is desired to reduce the standard deviation for repeatability to that indicated for reproducibility by *replicate* testing, then the number of tests required to yield *equal* variability may be determined by dividing the square of the repeatability by the square of the reproducibility, both at the same confidence level. Usually, this division will not result in a whole number. In this latter case, the number of tests required shall be the next larger whole number. This *reduced repeatability* figure applies only to the average of the number of tests indicated by the above calculation at the rating level employed. If a repeatability figure better than that for reproducibility is required, then the *desired* standard deviation figure is substituted for that shown for reproducibility before proceeding with the calculation as noted above to determine the number of replicate ratings whose average will have the desired degree of precision.

D 973 – 50,[3] Standard Method of Test for Butadiene Content of Polymerization Grade Butadiene: Revise as appended hereto.[7]

D 1020 – 52,[3] Standard Method of Test for Acetylene in Polymerization-Grade Butadiene (Silver Nitrate Method):

Title.—Change to read: "Acetylenes in Butadiene (Silver Nitrate Method)."

Section 1.—Change the first sentence to read: "This method describes a procedure for the determination of the monoalkyl acetylenes present in polymerization grade butadiene and concentrates in which butadiene is present in concentrations of 60 per cent or more."

New Section.—Add a new Section 7 entitled "Precision" to read as follows:

7. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent confidence):

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than the following:

Acetylenes, per cent by weight	Repeatability
0.20	0.02
0.50	0.15

(*b*) *Reproducibility.*—Results submitted by each of two laboratories shall not be considered suspect unless they differ by more than the following:

Acetylenes, per cent by weight	Reproducibility
0.10 or less	1.27 × mean
0.20	0.09
0.50	0.22

D 1024 – 53,[3] Standard Method of Test for Butadiene Dimer in Polymerization Grade Butadiene: Revise as appended hereto.[7]

D 1089 – 53,[3] Standard Method of Test for Carbonyl Content of Butadiene:

New Note.—Add a new Note 2 after Section 5, renumbering subsequent notes accordingly, to read: "NOTE.—Low results may be expected if the sample is not analyzed within 8 hr."

Section 7.—Change to read as follows:

7. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent confidence):

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than the following:

Carbonyl Concentration	Polymerization Grade Butadiene	60 per cent or More Butadiene
25 ppm	5 ppm	
1000 ppm	10 ppm	
		15 ppm

(*b*) *Reproducibility.*—Results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following:

Carbonyl Concentration	Polymerization Grade Butadiene	60 Per Cent or More Butadiene
25 ppm	5 ppm	
1000 ppm	10 ppm	
		70 ppm

D 1157 – 54,[3] Standard Method of Test for Total Inhibitor Content (*p*-Tertiary-Butyl-Catechol) of Butadiene:

Section 8.—Change to read as follows:

8. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent confidence):

(*a*) *Repeatability.*—Duplicate results obtained by the same operator should not be considered suspect unless they differ by more than 12 ppm.

(*b*) *Reproducibility.*—Results submitted by each of two laboratories should not be considered suspect unless they differ by more than 47 ppm.

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Method of Test for Color of Lubricating Oil and Petrolatum by Means of ASTM Union Colorimeter (**D 155 – 45 T**)[3] effective July 1, 1960, and its replacement by ASTM Method D 1500, Test for Measuring the Color of Petroleum Products (ASTM Color Scale).

In line with this action, editorial changes in Methods D 155 and D 1500 are necessary as recommended later in this report.

TENTATIVES CONTINUED WITHOUT REVISION

The committee has reviewed the following tentatives that have stood one or more years without revision and, for good and sufficient reason discussed within the committee, recommends their continuation as tentatives without revision:

- **D 240 – 57 T,** Test for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter
- **D 285 – 54 T,** Test for Distillation of Crude Petroleum
- **D 381 – 58 T,** Test for Existent Gum in Fuels by Jet Evaporation
- **D 445 – 53 T,** Test for Kinematic Viscosity
- **D 483 – 52 T,** Test for Unsulfonated Residue of Petroleum Plant Spray Oils
- **D 611 – 55 T,** Test for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents
- **D 721 – 56 T,** Test for Oil Content of Petroleum Waxes
- **D 808 – 57 T,** Test for Chlorine in Lubricating Oils and Greases by the Bomb Method
- **D 873 – 57 T,** Test for Oxidation Stability of Aviation Fuels (Potential Residue Method)
- **D 875 – 53 T,** Method of Calculation for Olefins and Aromatics in Gasoline
- **D 910 – 57 T,** Specifications for Aviation Gasolines
- **D 1018 – 55 T,** Test for Hydrogen in Petroleum Fractions by the Lamp Method
- **D 1085 – 57 T,** Method of Gaging Petroleum and Petroleum Products
- **D 1087 – 57 T,** Recommended Practice for Volume Calculations and Corrections in the Measurement of Petroleum and Petroleum Products
- **D 1160 – 57 T,** Test for Reduced Pressure Distillation of Petroleum Products
- **D 1263 – 53 T,** Test for Leakage Tendencies of Automotive Wheel Bearing Greases
- **D 1264 – 53 T,** Test for Water Washout Characteristics of Lubricating Greases
- **D 1317 – 57 T,** Test for Chlorine in Lubricating Oil (Sodium Alcoholate Volumetric Method)
- **D 1320 – 57 T,** Test for Tensile Strength of Paraffin Wax
- **D 1321 – 57 T,** Test for Needle Penetration of Petroleum Waxes
- **D 1368 – 58 T,** Test for Trace Concentrations of Tetraethyllead in Primary Reference Fuels
- **D 1405 – 57 T,** Test for Estimation of Net Heat of Combustion of Liquid Petroleum Products
- **D 1465 – 57 T,** Method of Test for Blocking Point of Paraffin Wax
- **D 1477 – 57 T,** Method of Test for Freezing Point of Aviation Fuels
- **D 1478 – 57 T,** Method of Test for Low-Temperature Torque of Ball Bearing Greases
- **D 1479 – 57 T,** Method of Test for Emulsion Stability of Soluble Cutting Oils
- **D 1480 – 57 T,** Method of Test for Density and Specific Gravity of Viscous Materials and Melted Solids by Bingham Pycnometer Method
- **D 1481 – 57 T,** Method of Test for Density and Specific Gravity of Hydrocarbon Liquids by Lipkin Bicapillary Viscous Oil Pycnometer

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following tentative and standards:

D 155 – 45 T,[3] Tentative Method of Test for Color of Lubricating Oil and Petrolatum by Means of ASTM Union Colorimeter:

Introductory Paragraph.—In the italicized boldface paragraph, replace the last sentence with: "Method D 155 will be withdrawn as an ASTM Tentative as of July 1, 1960."

D 1267 – 55,[3] Standard Method of Test for Vapor Pressure of Liquefied Petroleum Gases:

Appendix.—In Section A1, Paragraphs (*a*) and (*c*), change "2 ± $\frac{1}{16}$ in. in diameter" to read "2 ± $\frac{1}{8}$ in. in diameter." In Paragraph (*c*), in the next to last line, change "$\frac{9}{16}$ in. in diameter" to read "$\frac{1}{2}$ in. in diameter."

D 1500 – 58 T,[3] Tentative Method of Test for Measuring the Color of Petroleum Products (ASTM Color Scale):

Introduction.—Replace the last sentence with "Method D 155 will be withdrawn as an ASTM Tentative as of July 1, 1960."

D 323 – 58,[3] Standard Method of Test for Vapor Pressure of Petroleum Products (Reid Method):

None of the features of the method have been changed with respect to scope, apparatus, or procedure, but the format has been extensively revised.[7]

AMERICAN STANDARDS

The committee recommends the following for action by ASA Sectional Committee Z11 on Petroleum Products and Lubricants:

Approval as American Standard:

Methods of Analysis of Oil-Soluble Sodium Petroleum Sulfonates (D 855 – 56),
Method of Analysis of Calcium and Barium Petroleum Sulfonates (D 1216 – 56),
Method of Gaging Petroleum and Petroleum Products (D 1085 – 57 T),
Test for Total Inhibitor Content (p-Tertiary-Butyl-Catechol) of Butadiene (D 1157 – 59),
Test for Effect of Grease on Copper (D 1261 – 55),
Test for Lead in New and Used Greases (D 1262 – 55), and
Test for Mercaptan Sulfur in Jet Fuels (Amperometric Method) (D 1323 – 56).

Reapproval of American Standard:

Z11.8, Test for Water and Sediment by Means of Centrifuge (D 96 – 58 T),
Z11.10—1956, Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 – 56),
Z11.13—1952 Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method (D 129 – 58) (revised in 1958),
Z11.16—1948, Methods of Analysis of Lubricating Grease (D 128 – 57),
Z11.26—1958, Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158 – 54),
Z11.29—1935, Test for Dilution of Gasoline Engine Crankcase Oils (D 322 – 58 T) (revised in 1959),
Z11.37—1953, Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357 – 58),
Z11.43—1957, Test for Distillation of Plant Spray Oils (D 447 – 57 T),
Z11.47—1958, Test for Carbon Residue of Petroleum Products (Ramsbottom Coking Method) (D 524 – 58 T),
Z11.54—1947, Test for Ash Content of Petroleum Oils (D 482 – 46),
Z11.57—1949, Tests for Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils (D 810 – 48),
Z11.58—1949, Test for Sediment in Fuel Oil by Extraction (D 473 – 48),
Z11.66—1950, Test for Butadiene Content of Polymerization Grade Butadiene (D 973 – 50),
Z11.68—1957, Test for Sulfated Residue from New Lubricating Oils (D 874 – 57 T),
Z11.69—1958, Test for Knock Characteristics of Motor Fuels by the Research Method (D 908 – 58),
Z11.74—1952, Acetylene in Butadiene (D 1020 – 52),
Z11.79—1953, Test for Butadiene Dimer in Polymerization Grade Butadiene (D 1024 – 53), and
Z11.81—1953, Test for Carbonyl Content of Butadiene (D 1089 – 53).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[11]

This report has been submitted to letter ballot of the committee, which consists of 137 voting members; 92 members returned ballots, of whom 87 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. M. SMITH,
Chairman.

W. T. GUNN,
Secretary.

---

[11] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-3

ON

GASEOUS FUELS*

Committee D-3 on Gaseous Fuels held one meeting during the year: on October 12, 1958, in Atlantic City, N. J., in conjunction with the Annual Convention of the American Gas Assn.

The committee was advised of the death of W. R. Fraser, who had long represented the Operating Section of the American Gas Assn. on Committee D-3. Mr. O. W. Lusby was elected his successor.

Mr. H. B. McNichols was elected as chairman of Subcommittee II, and A. F. Cascioli of Subcommittee III. Mr. O. W. Lusby was elected chairman of Subcommittee V.

AMERICAN STANDARD

The committee recommends that the Standard Method for Measurement of Gaseous Fuel Samples (D 1071 – 55) be submitted to the American Standards Assn. for approval as American Standard.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Measurement of Gaseous Samples (H. B. McNichols, chairman) was requested to consider studies in progress by the ASME Research Committee on Fluid Meters dealing with measurement of small volumes. The current program covers orifice meter runs in pipe sizes of $\frac{1}{4}$ to 1 in. inclusive, to determine effects of orifice bore, orifice setting, and pipe roughness. A progress report is now being awaited.

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (A. F. Cascioli, chairman) has been studying the use of the recording calorimeter in gas heating value determinations. Plans have been initiated for incorporating into a standard test method studies by the American Gas Assn. on the Cutler-Hammer recording calorimeter when used with gases of high-heating value, and the work of the National Bureau of Standards in producing a standard 1000 Btu per cu ft gas.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. E. Stovall, chairman) is considering the numerous developments and improvements in equipment for measurement of specific gravity since preparation of the Standard Method D 1070, Test for Specific Gravity of Gaseous Fuels. Committee D-3 requested that the subject be reviewed in the light of existing conditions and any need for action in preparation of future standard methods reported for review and action.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (E. G. Hammerschmidt, chairman) considered suggestions from its chairman that other available instruments for water vapor determination be studied. It was suggested that future work should include a method covering a moisture recorder.

Subcommittee VII on Complete Analysis of Chemical Composition of Gaseous Fuels (D. V. Kniebes, chairman) is engaged in a revision of the Method for Analysis of Carbureted Water Gas by the Mass Spec-

---

*** Sixty-second Annual Meeting of the Society, June 21–26, 1959.**

trometer (D 1302 – 53 T). Attention has also been directed to the desirability of a chromatography method. Research is now in progress at the Institute of Gas Technology covering analysis of natural gas by this method for industry acceptance. Using the data from this study as a basis, it is planned to prepare a method for committee consideration. Additional members familiar with such analysis will be added to the membership of Subcommittee VII.

This report has been submitted to letter ballot of the committee, which consists of 35 members; 26 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. V. KNIEBES,
Chairman.

K. R. KNAPP,
Secretary.

REPORT OF COMMITTEE D-4
ON
ROAD AND PAVING MATERIALS*

Committee D-4 on Road and Paving Materials held two meetings during the year: in Boston, Mass., on June 25, 1958, and in Pittsburgh, Pa., on February 6, 1959.

The committee lost four members by death: Messrs. C. W. Allen, W. H. Fulweiler, A. E. Stoddard, and H. S. Mattimore. In recognition of the many valuable contributions of these members to the work of the committee and the loss which members felt in their passing, appropriate Memorial Resolutions were adopted and they are being entered on the permanent records of the committee and the Society.

The committee sponsored a symposium during the 1958 Annual Meeting on "The Effect of Water on Bituminous Paving Mixtures."[1] At the 1959 Annual Meeting, the committee is sponsoring symposia on "The Practical and Statistical Significance of Tests and Properties of Bituminous Paving Binders," and "Methods of Test on Design of Bituminous Paving Mixtures," as well as a general papers session which will include four technical papers.[2]

Plans are well under way to sponsor a committee session during the Third Pacific Area National Meeting to be held in San Francisco on October 11 to 16, 1959.[3]

A few subcommittees now have a rather large membership and the committee adopted an amendment to its by-laws limiting membership in subcommittees to 25 in number, including the chairman. The committee is also considering an amendment to its by-laws which would require a two-thirds affirmative vote by letter ballot of a subcommittee for approval of a recommendation, rather than a simple majority as at present.

During the year, the committee realigned the duties of its officers to spread more equitably the work formerly accomplished by the Secretary. A Membership Secretary was added to the group of officers, and it will be his duty to take care of all matters relating to membership in the committee and its subcommittees.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Committee D-4 concurs with Committee D-18 in the publication as tentative of the following methods which were accepted by the Administrative Committee on Standards on March 25, 1959:

Tentative Methods of Test for:

Making and Curing Soil Cement Compression and Flexure Test Specimens in the Laboratory (D 1632 - 59 T),
Compressive Strength of Molded Soil-Cement Cylinders (D 1633 - 59 T),
Compressive Strength of Soil-Cement Using

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

[1] Issued as separate publication *ASTM STP No. 240.*

[2] Issued as separate publication *ASTM STP No. 252.*

[3] To be issued as separate publication *ASTM STP No. 277.*

Portions of Beam Broken in Flexure, Modified Cube Method (D 1634 - 59 T), and
Flexural Strength of Soil-Cement Using Simple Beam with Third-Point Loading (D 1635 - 59 T).

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[4]

Tentative Methods of Test for:

Engler Specific Viscosity of Tar Products,
Coating and Stripping of Bitumen-Aggregate Mixtures, and
Penetration of Bituminous Materials.

Tentative Specifications for:

Hot-Mixed, Hot-Laid Asphalt Paving Mixtures.

REVISION OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Specifications for: Crushed Stone, Crushed Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 - 54):[5] Revise as appended hereto.[6]

Standard Specifications for Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 - 54):[5] Revise as appended hereto.[6]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Calcium Chloride (D 98 - 56 T).

Tentative Method of:

Test for Scratch Hardness of Coarse Aggregate Particles (C 235 - 57 T),[7] and
Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 - 58 T).

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 - 42):[5]

Section 1(a).—Delete the words "at room temperature."

Section 2(b).—Change the opening words to read "A wire basket of No. 6 or No. 8 mesh. . . ."

Section 3.—In the first sentence change the words "rejecting all material passing a $\frac{3}{8}$-in. sieve" to read "rejecting all material passing a No. 4 sieve." Delete the last sentence.

Section 4.—In Paragraph (*a*) delete "at 59 to 77 F (15 to 25 C)." In Paragraph (*b*) insert "at 68 to 77 F (20 to 25 C)" at the end of the last sentence.

Standard Method of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 - 57):[5]

Section 1(a).—Delete the words "at room temperature."

---

[4] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 4.

[5] 1958 Book of ASTM Standards, Part 4.

[6] The revised specifications appear in the 1959 Supplement to Book of ASTM Standards, Part 4.

[7] This method is under joint jurisdiction with Committee C-9, and the recommendation for adoption as standard has not as yet been approved by Committee C-9.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following methods:

Tentative Methods of Test for:

Loss on Heating of Oil and Asphaltic Compounds (**D 6 - 39 T**),
Vacuum Distillation of Liquid and Semi-Solid Asphaltic Materials to Obtain a Residue of Specified Penetration (**D 1189 - 52 T**), and
Moisture or Volatile Distillates in Bituminous Paving Mixtures (**D 1461 - 57 T**).

EDITORIAL CHANGE

The committee recommends an editorial change as follows in the Standard Methods of Sampling Bituminous Paving Mixtures (**D 979 - 51**):[5]

Table I.—In the first column change "No. 10" sieve to "No. 8."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[8]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A-4 on Papers (N. W. McLeod, chairman) has been active in the solicitation of suitable technical papers for presentation at the 1959 Annual Meeting of the Society. Four such papers are being presented at the current Annual Meeting.

Subcommittee B-2 on Physical Tests for Compressed Bituminous Mixtures (L. F. Rader, chairman) has been authorized by the Executive Subcommittee to prepare a "Manual of Recommended Practices for the Design of Bituminous Paving Mixtures." The manual will be a comprehensive separate publication in the Special Technical Publication (STP) series and will include the four methods of tests now standardized by ASTM, namely, the Hubbard-Field, the unconfined compression test, the Marshall test and the Hveem stabilometer and cohesiometer tests, together with sections on the preparation of aggregates for test and on density and voids. Mr. Rader is acting as chairman of a Symposium Committee on "Methods of Test for Design of Bituminous Paving Mixtures" which will provide papers for a session at the Annual Meeting.

Subcommittee B-3 on Distillation (J. W. Donegan, chairman) has found that the precision of Method D 402 can be improved by (1) changes in thermometers, and (2) calculation of distillate fractions as per cent of total sample instead of percentages by volume of total distillate. Cooperative testing is continuing in order to gather more data upon which to base a precision statement for the method.

Subcommittee B-5 on Softening Point (D. F. Fink, chairman) is concluding its cooperative testing program on the single thermometer/bath method and is preparing a revision of Method D 36. The single thermometer/bath method will give slightly different softening point results than Method D 36, but cooperative work has established correction factors from which conversion tables can be prepared. Studies of the precision of Method D 36 have been completed and a new precision statement will be proposed.

Subcommittee B-6 on Extraction and Recovery of Constituents from Bituminous Mixtures (E. W. Klinger, chairman) has five new methods of test under consideration: (1) recovery of asphalt from benzene solution by the Abson method; (2) bitumen content of paving mixtures by the improved method reflux extractor; (3) Maryland method for extracting bitumen from paving mixtures; (4) bitumen content of paving mixtures by reflux extraction; and (5) bitumen content of paving mixtures by immersion-reflux extraction.

---

[8] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee B-12 on Structural Properties of Mineral Aggregates (Stanton Walker, chairman) in a joint effort with Subcommittee III-e of Committee C-9 is studying Method C 131 for precision. In cooperative tests nine laboratories obtained abrasion losses ranging from 21.8 to 30.2 on the same aggregate. A report on this work is expected by the June meeting.

Subcommittee B-14 on Specific Gravity (H. T. Williams, chairman) is now developing a program for the preparation of a method of test for effective specific gravity of bituminous coated aggregates. There is a strong demand for such a test in connection with the design of bituminous paving mixtures.

Subcommittee B-15 on Sampling Road Materials (R. E. Britton, chairman) is recommending the adoption of Method D 75 as standard, as noted earlier in the report.

Subcommittee B-16 on Setting Qualities of Bituminous Materials (A. B. Cornthwaite, chairman) is completing work on the rolling-ball method for determining the curing rate of liquid asphalts.

Subcommittee B-17 on Emulsion Tests (P. E. McCoy, chairman) is continuing work on stone-coating water resistance tests for distinguishing between the slow curing SS types of emulsions and the medium setting MS types suitable for coarse aggregate mix work. Three task forces are at work on (1) stone-coating water resistance, (2) demulsibility, and (3) viscosity.

Subcommittee B-18 on Loss on Heating (W. E. Spelshouse, chairman) is completing cooperative work to determine the precision of Method D 6. Six asphalt samples have been tested by seven cooperators. Studies of the D 6 oven with respect to (1) effects of heat, (2) effects of size, and (3) effects of air flow will continue.

Subcommittee B-19 on Accelerated Tests for Durability of Bituminous Materials (D. C. Taylor, chairman) has two new methods of test under development: (1) method of test for determining the effect of heat and air on asphaltic materials, more commonly known as the Bureau of Public Roads Thin Film Test; and (2) method of test for an aging index of bituminous materials by use of the Shell sliding plate microviscometer. There is a marked correlation between the aging index determined by the Shell method and the per cent retained penetration as determined by the thin film test according to test data.

Subcommittee B-24 on Penetration Test (J. York Welborn, chairman) is continuing studies for the improvement of Method D 5.

Subcommittee B-26 on Effect of Water on Bituminous Coated Aggregates (W. H. Goetz, chairman) developed the new Tentative Method of Test for Coating and Stripping of Bitumen-Aggregate Mixtures referred to earlier in the report.

Subcommittee C-8 on Water-Bound Roads (J. E. Wood, chairman) is actively engaged in studies of both a research and test nature for possible modifications of Standard Specifications for Crushed Stone, Crushed Slag and Gravel for Water-Bound Macadam Base and Surface Courses of Pavements (D 694 – 55).

Subcommittee C-10 on Plant Mix Bituminous Surfaces and Bases (Fred Hubbard, chairman) has submitted the proposed revisions of Specifications D 692 and D 1073 referred to earlier in the report, and is consolidating Specifications D 947 and D 978 into a single specification. Work on the revisions was accomplished in collaboration with representatives of the American Association of State Highway Officials and The Asphalt Institute.

Subcommittee C-13 on Bituminous Paving Plant Operations (J. E. Ward, chairman) has been actively engaged in a review of standard Specification D 995.

Subcommittee D-1 on Calcium Chloride and Sodium Chloride for Use as Road Materials (H. F. Clemmer, chairman) is making further studies on Specifications D 632 with the view of making recommendations for further revisions.

Subcommittee D-2 on Highway Traffic Marking Materials (Paints, Insets, and Strips) (N. G. Smith, chairman) is continuing its work toward the development of a performance type specification for traffic paints.

Subcommittee D-3 on Joint Fillers and Asphalt Planks (D. O. Woolf, chairman) is giving consideration to possible changes in the Standard Specifications for Preformed Expansion Joint Fillers for Concrete (D 544 – 56 T). This subcommittee is also preparing specifications for cold materials for sealing joints and concrete pavements, and for hot poured jet-fuel-resistant sealer and cold applied jet-fuel-resistant sealer.

This report has been submitted to letter ballot of the committee, which consists of 174 members; 130 members returned their ballots, of whom 120 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. B. CORNTHWAITE,
Chairman.

J. M. GRIFFITH,
General Secretary.

REPORT OF COMMITTEE D-5
ON
COAL AND COKE*

Committee D-5 on Coal and Coke held one meeting during the year: in Boston, Mass., on June 26, 1958. Meetings of the Advisory Subcommittee were held in Boston, Mass., on June 24, 1958 and in Pittsburgh, Pa., on Feb. 3, 1959. Subcommittee meetings were held in Boston in June, 1958, and in Pittsburgh in February, 1959.

During the year the committee lost 11 members, 3 by death; 13 members were added making a total voting membership of 63, of whom 17 are classified as producer, 23 as consumer, and 23 as general interest members.

One new subcommittee, Subcommittee XXVII, American Group for ISO/TC 27 on Solid Mineral Fuels, was organized with W. H. Ode as chairman. Subcommittee XVIII on Classification of Coals was placed on inactive status.

ISO Activities.—Committee D-5, through the American Standards Assn., has continued to serve as the official USA group participating in the development of international standards being conducted by Technical Committee 27, Solid Mineral Fuels, of the International Organization for Standardization (ISO). The fifth meeting of ISO/TC 27 was held at Harrogate, England, June 12 to 18, 1958. The United States was represented by a delegation of four selected by Committee D-5 and approved by the American Standards Assn. as follows:

O. W. Rees, Leader,
C. C. Russell, Alternate Leader,
R. F. Abernethy, and
C. R. Montgomery.

Detailed reports of the meeting were prepared by Messrs. Russell, Abernethy, and Montgomery and placed in the Committee D-5 files as well as in the headquarters files of ASTM and ASA. A brief summary report was prepared by O. W. Rees for limited circulation. The USA delegates took active part in the deliberations at the meeting and, among other things, were successful in directing international attention to certain tests and methods commonly used in the United States such as the movable-wall and sole-heated oven tests for evaluating coking properties of coals, the Gieseler test for plastic properties, the tumbler test for assessing coke properties, and the adiabatic calorimeter for determining calorific value of coal. Successful prosecution of these tests as well as several others will require effective future participation by the United States. One of the most important items facing ISO/TC 27 is the development of reliable international sampling methods. Committee D-5 is making an effort to participate in formulating these sampling methods.

By way of further participation, W. L. Glowacki, Eastern Gas and Fuel Associates, represented the United States at a meeting of Working Group 8 on Coke Testing on January 26–27, 1959, in Paris, France. He has prepared a report of this meeting which has been placed in the files of Committee D-5, ASTM Headquarters, and ASA Headquarters.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

In order to meet better the increasing demands of participation in the work of ISO/TC 27, a new subcommittee, Subcommittee XXVII, American Group for ISO/TC 27 on Solid Mineral Fuels, was formed with W. H. Ode, U. S. Bureau of Mines, as chairman. Efforts are under way in this subcommittee to provide USA representation at coming (May 1959) meetings of Working Group 7 on Sampling; Subcommittee 1 on Coal Preparation: Terminology and Performance; and Subcommittee 2 on Brown Coal and Lignite. In addition to handling distribution of ISO documents, this subcommittee is planning and organizing to secure money and experimental data necessary to our effective participation.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Nomenclature and Definitions (C. H. Sawyer, chairman) held one meeting at Boston, Mass., June 24, 1958 at which the review of terms used in standards was continued. The subcommittee voted to recommend to Committee D-5 that definitions pertinent to a given standard should be included in each standard as a separate section. It was agreed to refer certain terms to appropriate subcommittees for consideration and to suggest to Committee D-5 that it make editorial changes in standards to define the term "moist" as equilibrium moisture instead of natural bed moisture.

Subcommittee VII on Pulverizing Characteristics of Coal (H. F. Yancey, chairman) held one meeting on Oct. 10, 1958, at Fort Monroe, Va., at which revision of the Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 51) was further considered. As a guide to further research on the method arrangements were made to make a statistical study of the precision of present data.

Subcommittee XV on Plasticity and Swelling of Coal (Michael Perch, chairman) held one meeting on Feb. 2, 1959 at Pittsburgh, Pa. The proposed Gieseler method for determining plastic properties of coal was completed and approved by the subcommittee.

A study was made of an electric furnace heating method for the determination of free swelling index as compared to gas heating used in Method of Test for Free-Swelling Index of Coal (D 720 – 57). While this work is not complete, it appears that such a method may be feasible and may have certain advantages.

Two task groups were formed within the subcommittee to develop methods for measuring expanding properties of coals during carbonization by means of the movable-wall test oven and the sole-heated test oven.

Subcommittee XX on Sampling and Fineness Test of Pulverized Coal (E. L. Rogers, chairman) held one meeting on June 25, 1959 at Boston, Mass. Testing of the proposed new sampling tip has been continued.

Subcommittee XXI on Methods of Analysis (R. F. Abernethy, chairman).—A method for determining sulfur in coal ash was prepared by Section A and, after a few minor editorial changes, will be submitted to subcommittee letter ballot.

The proposed revised procedure for the determination of volatile matter in sparking coals (Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 58)) has passed through several drafts without final approval. A special group was appointed and requested to prepare a new draft for consideration.

An interlaboratory study of methods for determining forms of sulfur and chlorine in coal was conducted. A statistical analysis of the results obtained indicated the need for further laboratory work.

A series of tests was made by several laboratories on the suitability of the induction combustion furnace for determining total sulfur in coal. Inconsistent results were obtained and it was recommended that further work is not justified at present.

Cautionary notes relating to the bomb calorimeter used in Methods D 271 in regard to weight of sample, pressure of oxygen, inspection of bomb and position of operator while firing the bomb, were approved by Section C for submission to letter ballot of the main subcommittee.

A proposed draft of a method for determining mineral carbon dioxide in coal was submitted to letter ballot of Section C. Certain objections were resolved in meeting, with the recommendation that the revised draft be submitted to letter ballot of Subcommittee XXI.

A revision of the profiles now shown in the present standard method of test for fusibility of coal ash in Methods D 271 was approved for letter ballot of the subcommittee. In addition further progress was made on the proposed revision of the method for determining fusibility of coal and coke ash.

Subcommittee XXII on Physical Tests of Coke (B. P. Mulcahy, chairman) in its meeting held in Pittsburgh, Pa., Feb. 4, 1959, considered further the proposed revision of the Method of Drop Shatter Test for Coke (D 141 – 48). Consideration was also given to a complete revision of Method D 167.

Subcommittee XXIII on Sampling (W. M. Bertholf, chairman).—Jan Visman, Canada Dept. of Mines, replaced R. L. Coryell as chairman of Section C on Sample Preparation.

Section A prepared a second draft of the proposed method for mechanical sampling of coal. This was revised through discussion in meeting and necessary further work arranged for. A first draft of the proposed method for determining variance components of coal was distributed.

A proposed method for preparing coal samples for analysis was approved for submission to Subcommittee XXIII for action pending approval of Section C by letter ballot.

Subcommittee XXVII, American Group ISO/TC 27 (W. H. Ode, chairman).—The first meeting of this new subcommittee was held in Pittsburgh, Pa., Feb. 3, 1959 at which ways and means for developing more effective participation in the work of ISO/TC 27 were considered. The way was cleared for organizing working sections within the subcommittee. Definite interest in this international activity was evidenced by the attendance at the meeting of more than 30 guests, some of whom took active part in the discussions.

W. L. Glowacki gave a report of the meeting held in Paris, France, referred to earlier in this report under ISO Activities.

This report has been submitted to letter ballot of the committee which consists of 63 voting members; 57 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

O. W. REES,
Chairman.

R. L. CORYELL,
Secretary.

REPORT OF COMMITTEE D-6
ON
PAPER AND PAPER PRODUCTS*

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 26, in Boston, Mass., and on February 27, in New York City.

The Advisory and other subcommittees held meetings in conjunction with the main committee. At the present time, the committee consists of 99 members, of whom 79 are voting members; 31 are classified as producers, 23 as consumers, and 45 as general interest.

Committee D-6 held a Symposium on Paper and Paper Products: New Developments with Accompanying Requirements for New Testing Methods. Six papers were read in two sessions during the Annual Meeting of the Society:[1]

"Some Historical Developments in Paper Testing," by W. R. Willets and F. R. Marchetti

"Testing of Synthetic Fiber Papers," by F. H. Koontz and J. K. Owens

"Technology of Non-Woven Structures: Paper and Textile Processes and End Uses," by J. T. Taylor and F. J. McLaughlan

"New Developments in the Internal Bonding of Paper," by K. W. Britt

"Polyethylene Extrusion Coated Papers—An Appraisal of Testing Methods," by K. Thompson

"Knit Paper—Its Uses as a Utilitarian Textile," by R. H. Marks

A Symposium on Relative Humidity, Its Measurement and Effect has been proposed, to be given during the Eleventh TAPPI Testing Conference, September 27 to 29, 1960, Grand Rapids, Mich.

REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[2] of the Tentative Method of Test for Puncture and Stiffness of Paperboard, Corrugated and Solid Fiberboard (**D 781 – 44 T**)[3] and continuation of the method as tentative.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 – 48):[3]

New Method.—Add a new Method B as appended hereto.[4] The present method (Sections 1 through 8) will remain as Method A.

Standard Methods of Test for Ply Separation of Combined Container Board (D 1028 – 51):[3]

New Section.—Add a new Section 2

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] These symposium papers were published as *ASTM STP No. 241.*

[2] The revised tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 6.

[3] 1958 Book of ASTM Standards, Part 6.

[4] The new method is included in the revised standard, and appears in the 1959 Supplement to Book of ASTM Standards, Part 6.

entitled "Significance" to read as follows, renumbering subsequent sections accordingly:

2. *Significance.*—The ability of a container to meet certain water-resistant requirements is influenced by the water resistance of the adhesive used in the lamination. Water resistance of the adhesive bond is related to strength and absorbency, and thus should be used as a criterion for water resistant paper containers.

Standard Method of Test for Peeling Resistance of Paperboard (D 1029 - 52):[3]

New Section.—Add a new Section 2 entitled "Significance" to read as follows, renumbering subsequent sections accordingly:

2. *Significance.*—The peeling resistance of paperboard is greatly influenced by the extent of pulp refining on the paper machine and the moisture content of the board at the time of combining on the corrugator. Although few liners fail in this manner, the failures that do occur result in poor quality board from an appearance and printability standpoint.

Section 5.—Change ASTM reference for conditioning of specimens from "Method of Conditioning Paper and Paper Products for Testing (ASTM Designation: D 685)" to "Method of Conditioning Paperboard, Fiberboard, and Paperboard Containers for Testing (ASTM Designation: D 641)."

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Method of Test for Dimensional Changes of Paper with Changes in Moisture Conditions (D 1270 - 53 T).[3]

EDITORIAL CHANGE

The committee recommends editorial changes as follows in the Standard Method of Test for Ash Content of Paper and Paper Products (**D 586 - 42**):[3]

Section 5.—Delete the last sentence and replace with the following: "The sample weight shall be of sufficient size to give an ash weight consistent with the required reproducibility as given in Section 8."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sampling and Conditioning (T. W. Lashof, chairman).—The Task Group on Sampling, working jointly with the Technical Assn. of Pulp and Paper Industry (TAPPI), is working on a revision of Method D 585 which will be published in two parts: a procedure for obtaining a representative sample of paper or paperboard for testing, and another procedure for determining the required sample size and statistical criteria for acceptance. Other joint task groups with TAPPI are investigating equipment for measuring relative humidity and making recommendations for interlaboratory investigations.

Subcommittee II on Chemical Methods (A. P. Tracy, chairman).—Task groups are actively studying mold resistance, qualitative and quantitative methods for fillers, ashing, and starch content. One method is ready for balloting.

Subcommittee III on Paper Testing (L. Price, chairman).—The Task Group on Basis Weight is completing its final report on this subject. The Task Group on Tensile Breaking Strength of Paper is preparing a final draft of the method for letter ballot. Other items undergoing work are flammability, bursting strength, and thickness of paper.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Physical Test Methods for Container Board (W. B. Lincoln, Jr., chairman).—Task groups are preparing final drafts on methods for tear and bursting strength of container board. The method of testing the flat crush of corrugated medium is receiving further study.

Subcommittee VI, Editorial (W. R. Willets, chairman).—Two significance statements have been prepared for inclusion in Methods D 1028 and D 1029.

Subcommittee VII, ISO/TC6 on Paper (L. S. Reid, chairman) will work jointly with TAPPI and other groups to act as an advisory group to participate in the activities of the ISO/TC6 Committee on Paper.

This report has been submitted to letter ballot of the committee, which consists of 80 voting members; 47 members returned their ballots, of whom 41 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. H. Carter,
Chairman.

R. E. Green,
Secretary.

REPORT OF COMMITTEE D-7
ON
WOOD*

Committee D-7 on Wood and ten of its subcommittees met at Madison, Wis., on January 28 to 30, 1959. Meetings of the following subcommittees preceded the main committee meeting on January 30: Subcommittee II on Laminated Timber; Subcommittee IV on Wood Paving Blocks; Subcommittee V on Methods of Preservative Treatment of Timber; Subcommittee VI on Timber Preservatives; Subcommittee VII on Wood Poles and Cross Arms; Subcommittee IX on Methods of Testing; Subcommittee X on Nomenclature and Definitions; Subcommittee XI on Moisture Content of Timber; Subcommittee XIII on Durability and Exposure; and Subcommittee XV on Wood-Base Fibers and Particle Panel Materials.

Special features of the 3-day meeting included a program on developments in forest products research, a tour of the Forest Products Laboratory with a number of special demonstrations, and an evening dinner meeting with an illustrated talk by L. J. Markwardt on the "International Exposition in Color" featuring the extensive wood construction employed in various buildings at Brussels.

Six new members were elected to the committee; there were four resignations. The death of W. H. Fulweiler, Chairman of Subcommittee XII on Fire-Retardant Wood and Past-President of ASTM, which occurred on December 20, 1958, was reported. Tribute was paid to Mr. Fulweiler for his long and continued work and interest in the committee.

The chairman announced with regret the resignation of L. W. Smith as secretary of the committee. A vote of thanks was extended for his services. William A. Oliver, Professor of Civil Engineering at the University of Illinois, was unanimously elected secretary in his place.

Committee D-7 is participating in the Third Pacific Area National Meeting of the Society through the development of two symposium programs, one on the general subject "Wood in Building Construction"[1] and the other on "Treated Wood for Marine Use."[1]

At the last meeting of the committee, the chairman reported on a number of International Standardization Organization committees relating to the field of wood and wood-base materials that have been organized in recent years. It was recalled that in 1951, the attention of the committee was directed to ISO Project, ISO/TC/55, Resinous Lumber, and at that time it was recommended that Committee D-7 be represented, at least on a liaison basis. The committee is kept in touch with the work of this committee through the ASA in its capacity as observer on all ISO committees in which there is no direct Amer-

---

*** Sixty-second Annual Meeting of the Society, June 21–26, 1959.**

[1] To be issued as separate technical publication *ASTM STP No. 275.*

ican participation. There are presently five approved ISO Projects in the field of forest products as follows: ISO/TC/55, Resinous Lumber; ISO/TC/92, Fire Tests of Building Material for Construction; ISO/TC/89, Fiber Building Board; ISO/TS/P40, Plywood and Particle Board; and ISO/TS/P41, Wood (Characteristics and Nomenclature). The committee has carried on an extended discussion of the ISO program in these several fields and the possibility and desirability of American participation. Participation would be considered if ways and means could be found of obtaining support for such action. The chairman will continue to obtain such information as is available on the progress of the several ISO committees through the ASA and keep the committee informed of any important progress of matters of interest.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee D-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Acid Copper Chromate (D 1624 – 59 T),
Chromated Copper Arsenate (D 1625 – 59 T), and
Chromated Zinc Arsenate (D 1626 – 59 T).

New Tentative Methods for:

Chemical Analyses of Acid Copper Chromate (D 1627 – 59 T),
Chemical Analyses of Chromated Copper Arsenate (D 1628 – 59 T), and
Chemical Analyses of Chromated Zinc Arsenate (D 1629 – 59 T).

These recommendations, with the exception of D 1626 and D 1629, were accepted by the Standards Committee on March 25, 1959. The remaining two tentatives were accepted by the Standards Committee on July 13, 1959.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Methods of Conducting Machining Tests of Wood and Wood-base Materials, as appended hereto.[2]

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specifications for Wood Paving Blocks for Exposed Platforms, Pavements, Driveways, and Interior Floors Exposed to Wet and Dry Conditions (D 52 – 58 T),[3] and continuation of the specification as tentative.

Section 3(b).—Revise to read as follows:

> (*b*) Blocks manufactured of southern yellow pine or Douglas fir shall average not less than four annual rings per inch when measured over the third, fourth, and fifth inches from the pith. In blocks not containing the pith, measurement shall be made on a 3-in. radial line over a portion of the cross-section that is representative of the average growth. No block shall have less than three rings per inch measured as above, and not more than 5 per cent of the pieces in any lot may have three rings per inch.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Test for Coke Residue of Creosote (D 168 – 30):[3] Revise as appended hereto.[4]

Standard Method of Test for Distillation of Creosote (D 246 – 49):[3] Revise as appended hereto.[4]

---

[2] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 6.

[3] 1958 Book of ASTM Standards, Part 6.

[4] The revised standards appear in the 1959 Supplement to Book of ASTM Standards, Part 6.

Standard Specifications for Creosoted End-Grain Wood Block Flooring for Interior Use (D 1031 - 55):[3]

Section 3.—Delete Paragraphs (*b*) and (*c*) and substitute a new Paragraph (*b*) to read as follows:

> (*b*) Blocks manufactured of southern yellow pine or Douglas fir shall average not less than four annual rings per inch when measured over the third, fourth, and fifth inches from the pith. In blocks not containing the pith, measurement shall be made on a 3-in. radial line over a portion of the cross-section that is representative of the average growth. No block shall have less than three rings per inch measured as above, and not more than 5 per cent of the pieces in any lot may have three rings per inch.

Standard Method of Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (D 1101 - 58):[3]

Title.—Revise the title to read: "Standard Method of Test for Integrity of Glue Joints in Structural Laminated Wood Products for Exterior Use."

Introduction.—Insert the word "structural" before "glued laminated members" in the first line, and before "laminated members" in the fifth line.

Section 1.—Insert the word "structural" before the words "laminated wood members."

Section 5(a).—In line 4 change "75 to 85 F" to read "65 to 80 F." In line 13 change "1 hr" to read "2 hr." In line 17 change "2½ hr" to read "4½ hr." In line 18 change "93½ hr" to read "91½ hr."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (L. W. Wood, chairman).—Study is currently under way on the possible need for revision of the Tentative Methods for Establishing Structural Grades of Lumber (D 245 - 57 T).

Subcommittee II on Laminated Timber (F. J. Hanrahan, chairman).—In addition to the revision of Method D 1101 reported elsewhere, much consideration has been given to ways and means of further reducing the time required for evaluating the integrity of glue joints. A 3-hr test applicable to oak has been developed by the Forest Products Laboratory in cooperation with the Navy Bureau of Ships that appears very promising and will be explored further. Further study is contemplated in the development of a reduced time method applicable to softwoods.

Much attention is being given to the need of providing a series of simple, practical tests for application in quality control programs. The subcommittee has made plans to prepare specifications and methods covering (*a*) a modification of the standard block shear test for testing plane scarf joints, (*b*) a tension test for finger and other than plane scarf joints, and (*c*) a simple bending test for rough testing of scarf joints. Other test methods will also be studied and considered during the coming year.

Subcommittee III on Plywood (J. A. Liska, chairman).—No immediate changes are contemplated in the present standards relating to plywood (Methods D 805 and Definitions D 1038), but the committee is looking ahead toward the possible development of test procedures involving large plywood specimens in addition to the coupon type now commonly employed.

Subcommittee IV on Wood Paving Blocks (W. H. O'Brien, chairman).—The principal work consisted in the revision of the two specifications for wood paving blocks (D 52 and D 1031) with respect to quality of the material. The changes establish modified rates of growth requirements for southern yellow pine and Douglas fir.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Methods of Preservative Treatment of Timber (C. W. Best, chairman).—A draft of a specification for the pressure treatment of timber products, intended to cover various species and types of material and applicable to each of the treating processes, is being correlated with American Wood-Preservers' Assn. (AWPA) specifications. It is planned to complete the specifications for adoption during the coming year.

Subcommittee VI on Timber Preservatives (D. L. Davies, chairman).—The subcommittee is recommending revisions in Methods D 168 and D 246 to bring these methods up to date and to correlate them with the current AWPA standards.

Subcommittee VII on Wood Poles and Cross Arms (R. P. A. Johnson, chairman).—The main activity of the subcommittee continues to be the conduct of the ASTM Wood Pole Research Program under the immediate direction and support of three task groups. The testing of all untreated and treated full size poles and the matched small, clear specimens in Program No. 1 have been completed, and twelve interim reports have been issued. The interim reports presented the test results and associated information, species by species, but did not present conclusions. General analysis of data is now under way, and work on the final report has begun. The final report will present a summary of the data and a complete analysis of the results, together with conclusion and recommendations. Included also will be related data from other studies of importance in wood pole design and specifications. The report will be particularly pertinent to the work of the ASA Sectional Committee on Specifications for Wood Poles, ASA Project O5. Testing is complete, and a report is in preparation on treated longleaf pine poles and matched small, clear specimens in Program No. 2 to show the effects of modified conditioning schedules on treatment and strength. On the financial side, the subcommittee reports a total receipt and expenditures of approximately $300,000 on Programs I and II. This affords an indication of the scope and importance of this cooperative research effort.

Subcommittee IX on Methods of Testing (L. J. Markwardt, chairman).—The principal accomplishment of the subcommittee during the year has been the development of the proposed Methods of Conducting Machining Tests of Wood and Wood-Base Materials referred to earlier in the report.

Problems relating to the sawing and machining of wood are of international interest and importance. The Conference on Wood Technology of the Food and Agriculture Organization of the United Nations has had for some years an active Working Party on Sawing and Machining.

The subcommittee also has under consideration the development of a method of test for evaluating the strength and performance of joist hangers and similar devices used in conjunction with wood construction. Consideration is also being given to the preparation of a method of test for determining the bearing strength of metal connectors used in timber construction, based on procedures already used in extensive research.

Subcommittee X on Nomenclature and Definitions (R. R. Cahal, chairman).—The subcommittee has under consideration the possible revision of the Standard Definitions of Terms Relating to Timber (D 9 – 30).

Subcommittee XI on Moisture Content of Timber (R. C. Rietz, chairman).—

The subcommittee is presently concerned with various problems and techniques involved in determination of moisture content of wood. The subcommittee agreed to direct its activities toward (1) the development of moisture tests suitable for and involving laboratory techniques and (2) the development of testing methods that are nondestructive and are adaptable to industry application.

It is planned to develop a proposed tentative during the year to cover oven-drying, distillation, electrical, and hygrometric methods of determining moisture content.

Subcommittee XII on Fire-Retardant Wood (H. O. Fleischer, chairman).—Current interest in fire-retardant treatments for wood and wood-base materials and developments of fire test methods have brought out a number of items for study and consideration by the subcommittee. The development of specifications for fire-retardant treatments will be given attention during the year, and studies of fire test methods and reproducibility of results will be undertaken.

International activity in the field of fire test methods and research on fire-retardant treatments is reflected by the establishment of the International Organization for Standardization Committee ISO/TC/92, Fire Tests of Building Materials for Construction, on which ASA holds observer status and on which there is, as yet, no active American participation. In addition, the Food and Agriculture Organization of the United Nations (FAO) Conference on Wood Technology has an active working party on fire test methods for wood and wood-base materials on which the chairman of D-7 is a member. At the Madrid meeting of the FAO Conference last year, a special Symposium on Fire Test Methods was held with guest specialists from a number of countries in attendance. A report on the small tunnel furnace test for measuring surface flammability developed by the Forest Products Laboratory was presented. The conference recommended close cooperation with other international organizations working in the field of fire testing, namely, the International Council for Building Research Studies and Documentation and the ISO.

Subcommittee XIII on Durability and Exposure (R. M. Lindgren, chairman).—Continued study is being given to the requirements for the laboratory weathering procedure in Method D 1413 from the standpoint of correlation with outdoor weathering. The subcommittee is also preparing a new method of testing wood preservatives by field tests with stakes.

Subcommittee XIV on Methods of Chemical Analysis (W. E. Moore, chairman).—The subcommittee, in addition to some possible revision of present tentatives under its jurisdiction, contemplates the preparation of a tentative method for charcoal analysis during the coming year.

Subcommittee XV on Wood-Base Fibers and Particle Panel Materials (W. C. Lewis, chairman).—At its recent meeting, the subcommittee discussed the question of participation in ISO activities. In 1957, Committee D-7 recommended that the ISO proposal to establish a committee on Fiber Building Boards be voted negatively. However, this ISO committee was subsequently established as ISO/TC/89. While the subcommittee does not plan to participate actively in the work of ISO/TC/89 on an International Standard for Fiber Building Board, the chairman has been requested to keep the membership informed of the progress being made through information obtained by ASA in its status as observer.

A special task force of the subcommittee is continuing to study the question of nomenclature and is also reviewing Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials (D 1554 - 58 T) from the standpoint of possible additions and revisions.

The subcommittee is considering the need for test methods to evaluate the properties of finished fiberboard or particle board for flooring. A special task force was appointed to study the development of test methods and a product specification for hardboard. In addition, a special task force is continuing to survey the needs for a specification for a particle board, while another task force has been appointed to appraise the needs of activity on the new higher than usual density insulation board which is manufactured primarily for use as a structural material rather than thermal insulation.

The need for additional test methods on fiberboards and particle boards to supplement Methods D 1037 is recognized. At present, procedures for falling ball impact, abrasion, and direct screw withdrawal have been drawn up and reviewed by the subcommittee. In the discussion of the compression parallel to the surface test, it was brought out that a single method does not appear to be adequate. A paper summarizing experience obtained on several different methods on the range of fiber boards and particle boards is to be prepared for publication as a background.

This report has been submitted to letter ballot of the committee, which consists of 112 voting members; 57 members returned their ballots, of whom 54 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. J. MARKWARDT,
Chairman.

W. A. OLIVER,
Secretary.

REPORT OF COMMITTEE D-8
ON
BITUMINOUS MATERIALS FOR ROOFING, WATERPROOFING, AND RELATED BUILDING OR INDUSTRIAL USES*

Committee D-8 on Bituminous Materials for Roofing, Waterproofing, and Related Building or Industrial Uses held two meetings during the year: in Boston, Mass., on June 27, 1958, and in Pittsburgh, Pa., on February 3, 1959.

The membership consists of 80 voting members of whom 50 are classified as producers, 11 as consumers, and 19 as general interest members.

The committee suffered loss through death of W. H. Fulweiler.

A new subcommittee is being formed to develop standards for industrial pitches.

All the standards and tentatives for which the committee is responsible have been reviewed and recommendations concerning them are set forth in this report.

NEW TENTATIVES

The committee recommends for publication as tentative the following specification and methods of tests as appended hereto:[1]

Tentative Specification for:

Woven Glass Fabrics Saturated with Bituminous Substances for Use in Waterproofing.

Tentative Methods of:

Preparation of Test Panels for Accelerated and Outdoor Weathering of Bituminous Coatings, and

Test for Failure Endpoint in Accelerated and Outdoor Weathering of Bituminous Materials.

REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[1] of the Tentative Recommended Practice for Accelerated Weathering Test of Bituminous Materials (D 529 – 39 T)[2] and continuation of the recommended practice as tentative.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

Standard Specifications for Asphalt Shingles Surfaced with Mineral Granules (D 225 – 58):[2]

Table I.—Decrease the weight (net average of packages inspected) of finished shingles per 100 sq ft for type II shingles from 90.0 to 86.7 lb.

Decrease the weight of roofing per 100 sq ft (individual package) for type II shingles from 87.0 to 83.8 lb.

Decrease the weight of the exposed area per 100 sq ft for type II shingles from 87 lb to 83.8 lb.

Decrease the weight of the unexposed area per 100 sq ft for type II shingles from 87 lb to 83.8 lb.

Decrease the weight of weather side coating per 100 sq ft for type II shingles from 23.5 lb, to which it had recently been raised, to 18.5 lb.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new and revised tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

Standard Specifications for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (D 250 – 56):[2]

Section 9.—Add the following tensile strength requirements to the table of physical requirements:

	15-lb Type	30-lb Type
Minimum breaking strength at 77 F (25 C):		
With fiber grain, lb....	20	40 (plain only)
Across fiber grain, lb..	10	20 (plain only)

Standard Specifications for Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs (D 655 – 47):[2]

Eliminate the 20-lb type from the specifications, rewording where necessary.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of:

Sampling and Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing D 146 – 58 T).[2]

Tentative Specifications for:

Asphalt Insulating Siding Surfaced with Mineral Granules (D 1226 – 58 T),[2]

Asphalt-Base Emulsions for Use as Protective Coating for Built-Up Roofs (D 1227 – 52 T),[2] and

Woven Burlap Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 1327 – 57 T).[2]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (D 228 – 57 T).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 80 members; 56 returned their ballots, of whom 53 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. R. SNOKE,
Chairman.

G. W. ROBBINS,
Secretary.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-9
ON
ELECTRICAL INSULATING MATERIALS*

Committee D-9 held three meetings during the year: in Boston, Mass., during the Annual Meeting of the Society; in Philadelphia, Pa., on October 29, 30, and 31, 1958; and in Washington, D. C., on February 16, 17, and 18, 1959.

At the time of the Washington meeting of the committee, the membership comprised 225, of whom 104 were classified as producers, 73 as consumers, and 48 as general interest members.

On January 18, 1959, E. A. Snyder died; he was former chairman of Subcommittee IV and Honorary Member of Committee D-9.

The major organizational change during the year was the withdrawal of Subcommittee IV to form the new Committee D-27 on Electrical Insulating Liquids and Gases. Other organizational changes are: changes in the scope of the committee to take account of the withdrawal of Subcommittee IV; revisions in, or new titles and scope statements for, Subcommittees VI, VII, X, and XV to define clearly their fields of work; and formation of a new Subcommittee XIII on Composite Insulating Materials, which is still in the organizational stage.

Committee D-9 has continued to work with other groups in the field of electrical insulation such as the International Electrotechnical Commission (IEC), American Groups for the International Organization for Standardization (ISO), the American Institute of Electrical Engineers (AIEE), and the American Standards Association (ASA). The committee was represented on August 14, 1958, in Chicago, Ill., at the Joint Insulation Resistance Standards Meeting, which was sponsored by the Electronic Industries Association (EIA). Other cooperative activity consisted of appointments of representatives on the Pressure Sensitive Tape Council, on Subcommittee XVIII of Committee D-20, on Subcommittees 3 and 9 of Committee E-1, and on Subcommittee II of Committee F-1.

Two symposia, both sponsored by Subcommittee XII on Electrical Tests, were held during the year: "Factors Affecting Dielectric Breakdown Measurements," on Oct. 28, 1958, during the meeting in Philadelphia; and "Electrostatic Phenomena," on Feb. 17, 1959, during the meeting in Washington. Also, during the meeting in Philadelphia, the committee gave support to the International Symposium on Plastics Testing and Standardization, Oct 30 and 31, 1958, which was sponsored jointly by ASTM Committee D-20 and Technical Committee 61 on Plastics of the International Organization for Standardization (ISO/TC 61).[1]

With the exception of Recording Secretary, officers elected by Committee D-9 for the two-year term 1958–1960 are stated in the 1958 Annual Report of Committee D-9. Unforeseen difficulty in the election of a Recording Secretary made it necessary for J. E. Gibbons to continue in that office until October 31, 1958, at which time Thomas Hazen was elected to be Recording Secretary.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Published as separate publication *ASTM STP No. 247.*

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Methods of Testing:

Polymerizable Embedding Compounds Used for Electrical Insulation (D 1674 – 59 T), and

Flexible Vinyl Plastic Coated Sleeving Used for Electrical Insulation (D 1678 – 59 T).

Revisions of Tentative Methods of:

Test for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 – 59 T),

Testing Vulcanized Fibre Used for Electrical Insulation (D 619 – 59 T), and

Test for Dielectric Constant and Dissipation Factor of Polyethylene by Liquid Displacement Procedure (D 1531 – 59 T).

Tentative Revisions of Standard Methods of:

Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 58).

These recommendations were accepted by the Standards Committee on June 22, 1959, with the exception of the tentative revision of D 618 which was approved July 13, 1959. The new tentatives, revisions of tentatives, and tentative revisions of standards, excepting those referring to insulating liquids, will appear in the 1959 Compilation of ASTM Standards on Electrical Insulating Materials.

AMERICAN STANDARDS

The following standards under the jurisdiction of Committee D-9 have been approved as American Standard by the American Standards Association:

Specifications for:

Silicone Varnished Glass Cloth and Tape for Electrical Insulation (ASTM D 1459 – 57 T; ASA C59.38:1958).

Methods of:

Testing Varnishes Used for Electrical Insulation (ASTM D 115 – 55; ASA C59.30:1958),

Testing Varnished Cotton Fabrics and Varnished Cotton Fabric Tapes Used for Electrical Insulation (ASTM D 295 – 58; ASA C59.31:1958),

Test for Product Uniformity of Phenolic Laminated Sheets (ASTM D 634 – 44; ASA C59.32:1958),

Measuring Dimensions of Rigid Tubes Used for Electrical Insulation (ASTM D 668 – 52; ASA C59.33:1958),

Measuring Dimensions of Rigid Rods Used for Electrical Insulation (ASTM D 741 – 52; ASA C59.34:1958),

Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used for Electrical Insulation (ASTM D 902 – 56; ASA C59.35:1958), and

Testing Silicone Insulating Varnishes (ASTM D 1346 – 57; ASA C59.36:1958).

The committee recommends the following for action by ASA Sectional Committee C59 on Electrical Insulating Materials:

Approval as American Standard:

Specification for Absorbent Laminating Paper for Electrical Insulation (D 1080 – 58 T).

SUGGESTED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Suggested Method for Evaluation of Thermal Stability of Electrical Insulating Coated Sleevings (Dielectric Breakdown Method) as appended hereto.[2]

There is a need for this type of test and publication as a suggested method makes it possible to get early exploratory data, comments, and criticism, from which a decision to publish as a tentative method can be made sooner than otherwise possible.

---

[2] This suggested method appears in the 1959 Compilation of ASTM Standards on Electrical Insulating Materials.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods and recommended practice as appended hereto:[3]

Method of Measuring Absorbed Gamma Radiation Dose by Fricke Dosimetry:

This method of test was prepared by joint D-9/D-20 Subcommittee II on Effects of Radiation to provide a standard procedure for measurement of radiation dose absorbed in irradiation experiments.

Recommended Practice for Exposure of Polymeric Materials to High Energy Radiation:

This was prepared by joint D-9/D-20 Subcommittee II to provide accepted standardized procedures for exposure of plastics and organic electrical insulating materials to radiation in irradiation experiments designed to evaluate the effects of high energy radiations on properties.

Methods of Test for Dielectric Constant and Dissipation Factor of Expanded Cellular Plastics Used for Electrical Insulation:

These methods provide standardized procedures for measuring dielectric properties of foamed plastics in sheet form in thicknesses up to two inches.

Methods of Testing Electrical Grade Polytetrafluoroethylene Tubing:

These methods provide industry with standardized procedures for testing this type of tubing when intended for use as electrical insulation.

Methods for Sampling and Testing Untreated Mica Paper Used for Electrical Insulation:

These methods provide industry with standardized procedures for evaluating mica papers for electrical uses.

Methods of Test for Frictional Characteristics of Enameled Magnet Wire for Use in Winding Filled Coils:

These methods provide the electrical industry with methods for determining whether enamel coated magnet wire has sufficient lubricity to permit satisfactory winding of coils with cellulose acetate sheet interleaved between layers of wire.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Orange Shellac and Other Indian Lacs for Electrical Insulation (D 784 - 52 T):[4] Revise as appended hereto.[3]

This revision is being recommended to bring the specifications into accord with current needs and also to change the grade designations to correspond with those in Committee D-1's Standard Specifications for Orange Shellac and Other Lacs (D 237 - 57).

Tentative Methods of Testing Pressure-Sensitive Adhesive Coated Tapes Used for Electrical Insulation (D 1000 - 58 T):[4] Revise as appended hereto.[3]

This revision provides a method for flagging, to apply to class 2 tapes only. The purpose of this addition is to provide means of determining whether such properties as thickness, stiffness, and adhesion have been properly balanced for applications where flagging may occur.

---

[3] The new and revised tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 9.

[4] 1958 Book of ASTM Standards, Part 9.

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions, as appended hereto,[5] of the Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 58).[4]

This revision was prepared by joint D-9/D-20 Subcommittee XIV on Conditioning to provide nomenclature for conditioning prior to test and for test conditions similar to that used in Section 12 of Specifications D 709. The new nomenclature will permit shortening and simplification of the descriptions of conditioning procedures.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors (D 748 – 54 T),

Nonrigid Polyvinyl Tubing (D 922 – 54 T) with an editorial change in title to read "Specifications for Nonrigid Vinyl Plastic Tubing," and

Silicone Varnished Glass Cloth and Tape for Electrical Insulation (D 1459 – 57 T).

Tentative Methods of:

Test for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 – 59 T). (The adoption of this method is in a sense a recommendation for immediate adoption and requires a nine-tenths vote.)

Testing Solid Filling and Treating Compounds Used for Electrical Insulation (D 176 – 56 T),

Testing Pasted Mica Used in Electrical Insulation (D 352 – 56 T), and

Test for Dissipation Factor and Dielectric Constant Parallel with Laminations of Laminated Sheet and Plate Insulating Materials (D 669 – 42 T).

Tentative Recommended Practice for:

Cleaning Plastic Specimens for Insulation Resistance Testing (D 1371 – 55 T).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of all other tentatives under its jurisdiction.

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 58):[4]

Section 1.—In the second sentence delete the words "it is necessary to standardize the temperature, as well as the humidity conditions" and replace with "it is necessary to standardize the humidity conditions, as well as the temperature." This change is recommended in order to emphasize the importance of humidity conditioning.

The recommendations in this report have been submitted to letter ballot vote of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (W. E. Harvey, chairman) recommends the revision of Specifications D 784 referred to earlier in the report.

Current activities include studies on methods of determining gel time of laminating varnishes; on dry time, drainage, and dielectric strength of high-temperature varnishes; and on the resistance of insulating varnishes to refrigerants. Search continues for a satisfactory method for measuring viscosity of varnish, and a task group has in preparation a method for using the Brookfield viscometer. The task group will also consider the Ford cup, the bubble tube, and the Hoeppler viscometer. Mr. S. E. Berger, chairman of the task group, has been appointed to represent the subcommittee on Subcommittee 9 on Rheology of Committee E-1 on Methods of Testing.

---

[5] The tentative revision of the standard methods appears in the 1959 Supplement to Book of ASTM Standards, Part 9.

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Effects of Radiation (D. S. Ballantine, chairman) recommends adoption of one new tentative method and one new tentative recommended practice concerning nuclear radiation, as stated earlier in the report.

The subcommittee has continued its educational activities in the field of radiation phenomena. Lectures by subcommittee members were given on radioactivity during the 1958 Annual Meeting in Boston, and on dosimetry during the joint D-9/D-20 meetings in Washington in February, 1959. A symposium is in preparation on "Postirradiation Effects in Polymers." [7]

Subcommittee III on Plates, Tubes, Rods and Molded Materials (E. A. Russell, chairman) recommended the revision of Methods D 619 accepted by the Administrative Committee on Standards. The subcommittee also recommends adoption of the new Tentative Methods of Test for Dielectric Constant and Dissipation Factor of Expanded Cellular Plastics Used for Electrical Insulation.

Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 – 38), heretofore under joint jurisdiction of Committees D-9 and D-20 on Plastics, has been transferred to sole jurisdiction of Committee D-20 on Plastics.

Section J on Plastic Extruded Tubing has been transferred to Subcommittee VII on Flexible Sheet, Tape, and Tubing, as being more within the scope of that subcommittee's activity.

Other subcommittee activity of general interest includes participation in round-robin testing of insulation resistance of copper-clad laminates and further revision of Methods D 229 to provide for measurement of dielectric strength parallel to laminations.

Subcommittee IV on Liquid Insulation (F. M. Clark, chairman) held its last meeting as a subcommittee of Committee D-9 at the meeting in Washington in February. This report of subcommittee activity includes only the activities of the subcommittee which received Committee D-9 approval prior to the Washington meeting. Subsequent activities of the subcommittee will be reported in a separate Annual Report of Committee D-27, the successor to Subcommittee IV.

Subcommittee V on Ceramic Products (A. H. Scott, chairman) made no recommendations affecting standards during the year, but continued its survey of methods and specifications under its jurisdiction.

The subcommittee has formed a task group to review Methods C 328 for flexural properties and C 329 for specific gravity of fired ceramics. These methods are under the jurisdiction of Committee C-21 on Ceramic Whitewares and Related Products but are also used in electrical insulation.

Other subcommittee activity includes a review of a proposed tentative method for determination of the tensile strength of fired ceramic whiteware, which was prepared by Committee C-21, and a revision of Methods D 116.

Subcommittee VI on Solid Filling, Treating, Encapsulating, and Embedding

---

[7] To be issued as separate publication *ASTM STP No. 276.*

Compounds (Thomas Hazen, chairman) was reorganized under the direction of its chairman. A new subcommittee title and scope were approved. The new title is as given here, and the new scope statement reads as follows: The preparation of specifications and test methods for solid compounds applied as liquids by (1) melting and pouring (or dipping) or (2) pouring (or dipping) and subsequent polymerization.

The subcommittee recommended the new Tentative Methods of Testing Polymerizable Embedding Compounds Used for Electrical Insulation, accepted by the Administrative Committee on Standards. The subcommittee is also recommending adoption of Methods D 176 as standard without revision, as stated earlier in the report.

In the reorganized subcommittee, a new Section A on Fusible Compounds was formed.

Subcommittee VII on Flexible Sheet, Tape, and Tubing (F. P. West, chairman) was reorganized slightly during the year because of the transfer to the subcommittee from Subcommittee III of Section J on Plastic Extruded Tubing. A new subcommittee title was approved, as given here; and a new scope statement was adopted, which reads: The preparation of methods of test and formulation of specifications for flexible treated and untreated fabric and film in the form of sheet and tape, treated sleeving, and plastic tubing used for electrical insulation.

The subcommittee recommended the new Tentative Method of Testing Flexible Vinyl Plastic Coated Sleeving Used for Electrical Insulation accepted by the Administrative Committee on Standards. The subcommittee is recommending a new Suggested Method for Evaluation of Thermal Stability of Electrical Insulating Coated Sleevings (Dielectric Breakdown Method); a new Tentative Method of Testing Electrical Grade Polytetrafluoroethylene Tubing; a revision of Methods D 1000 to include a test for flagging of class 2 pressure sensitive adhesive tapes; and adoption as standard without revision of Specifications D 922 and D 1459, as listed earlier in the report. A minor editorial change is recommended in the title of Specifications D 922.

In progress in the subcommittee are a revision of a proposed specification for black and yellow straight cut and bias cut varnished cloth and tape, and revisions of Methods D 902 and D 1458.

L. J. Timm has been appointed to represent the subcommittee on Subcommittee XVIII on Reinforced Plastics of Committee D-20.

Subcommittee VIII on Insulating Papers (H. A. Anderson, chairman) made no recommendations affecting standards during the year. The subcommittee is considering, however, revision of Methods D 202 of sampling and testing untreated paper to add new test procedures; revision of Specifications D 1080 covering absorbent laminating paper to include cotton linter high absorbency paper for phenolic laminates; and revision of Specification D 1305 to include low density machine finish and high density water finish papers, in order that the specification will cover more adequately the grades of paper in commercial use. As the result of tests during the year, it now appears fairly certain that a reproducible life test can be included in insulating paper specifications.

The subcommittee has decided to postpone publication as tentative of the Proposed Specifications for Electrical Insulating Paper Sulfate (Kraft) Tissue for Capacitor Dielectric. The proposed specification was first published in the 1957 Annual Report of Committee D-9 and also as Appendix III of the September 1957 Compilation of ASTM

Standards on Electrical Insulating Materials.

The subcommittee has recommended Specifications D 1080 for approval as American Standard, as noted earlier in the report.

Subcommittee IX on Mica Products (K. G. Coutlee, chairman) recommends approval of a new Tentative Method for Sampling and Testing Untreated Mica Paper Used for Electrical Insulation, and adoption as standard without revision of Specifications D 748 and Methods D 352, as listed earlier in the report.

Work is in progress on thermal stability of the electrical properties of bonded flexible mica; and favorable comment has been received on the mica standards prepared by the subcommittee for use of the U. S. General Services Administration.

The subcommittee was represented by Harold Brafman at the ISO meeting in Harrogate, England, during the summer of 1958; and K. G. Coutlee has been appointed to represent the subcommittee on Subcommittee II on Insulators of Committee F-1 on Materials for Electron Tubes and Semiconductor Devices.

Subcommittee X on Magnet Wire Insulation (K. N. Mathes, chairman) recommends approval of the new Tentative Methods of Test for Frictional Characteristics of Enameled Magnet Wire for Use in Winding Filled Coils, as listed earlier in the report.

A statement of the scope of the subcommittee has been approved by Committee D-9, as follows: The preparation of methods of test for mechanical, electrical, chemical and thermal properties of magnet wire insulation and development of appropriate specifications. Coordination of activities with those of other groups is emphasized.

Subcommittee XI on Nomenclature, Significance and Statistics (C. L. Craig, chairman) has in preparation two recommended practices, one covering sampling procedures, and one on round-robin evaluation.

The subcommittee has made recommendations for changes in definitions in ASA Project C42, Definitions of Electrical Terms, through A. H. Scott, the Society's representative on C42.

A. R. Blanck has been appointed to represent the subcommittee on Subcommittee 3 on Elastic Strength of Materials of Committee E-1 on Methods of Testing.

Subcommittee XII on Electrical Tests (N. W. Edgerton, chairman) recommended the revisions of Methods D 149 and D 1531, accepted by the Administrative Committee on Standards, and now recommends adoption of Methods D 149 as standard without revision, as stated earlier in the report.

Work is in progress on dielectric loss and absorption at very high resistivities by the loss of charge method; and on the electrical testing of conductive rubber materials, to be added to Method D 257.

A. H. Scott reported publication of IEC Recommended Practice No. 93 for surface and volume resistivity of insulating materials. Recommended procedures are similar to those of Methods D 257. In progress in the IEC Working Group 2 of TC/15 are studies of insulation resistance using the tapered pin and strip electrodes of Methods D 257; of conditioning for resistivity tests, including a general document on conditioning; and of cells for liquid insulation. Other IEC studies are in progress on the heat resistance of solid insulation, on thermal endurance of magnet wire, and on a guide for preparing test specimens for examination. The last two of these three studies have advanced to the point of receiving IEC approval under its six-month rule.

The subcommittee's method for corona measurement, published as a Suggested

Method in the 1957 Annual Report of Committee D-9 and also as Appendix IV of the September 1957 compilation of methods and specifications, has been recommended for withdrawal for revision.

Subcommittee XIV on Conditioning (G. M. Armstrong, chairman), a joint D-9/D-20 subcommittee, recommended the tentative revision of Methods D 618 accepted by the Administrative Committee on Standards. The purpose of this revision was to bring temperature and humidity tolerances into agreement with recommendations accepted by ISO/TC 61 and IEC/TC 15, and to clarify practical meaning of the tolerances.

The subcommittee is recommending for approval a further tentative revision in Methods D 618 and an editorial change in the scope statement, as listed earlier in the report.

Subcommittee XV on Research and Technical Papers (H. S. Endicott, chairman) cooperated with Subcommittee XII in presentation of the two symposia mentioned earlier in this report and is now assisting in the preparation of a program for the Pacific Area National Meeting to be held in San Francisco during October 11 through 16, 1959.

The subcommittee is cooperating with Subcommittees III and XII in research at the University of Delaware on measurement of the insulation resistance of copper-clad laminates.

A scope statement, descriptive of the present activities of the subcommittee, has been approved by the Advisory Committee of Committee D-9, as follows: Promotion of studies on fundmental properties of electrical insulating materials and on their applications; and scheduling of symposia and solicitation of papers.

This report was compiled by the recently retired Recording Secretary, J. E. Gibbons, whose efforts are hereby gratefully acknowledged. Before submission, the report was reviewed by the present Recording Secretary, whose name appears below.

This report has been submitted to letter ballot of the committee, which consists of 275 members; 173 members returned their ballots, of whom 144 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. K. GRAVES,
Chairman.

THOMAS HAZEN,
Recording Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Methods of:

Test for A-C Capacitance, Dielectric Constant, and Loss Characteristics of Electrical Insulating Materials (D 150 – 54 T), and

Testing Fully Cured Silicone Rubber Coated Glass Fabric and Tapes for Electrical Insulation (D 1458 – 57 T).

Revision and Reversion to Tentative of Standard Methods of Testing:

Electrical Porcelain (D 116 – 44).

Tentative Revision of Standard Methods of:

Test for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149 – 59),

Testing Sheet and Plate Materials Used for Electrical Insulation (D 229 – 58),
Test for Electrical Resistance of Insulating Materials (D 257 – 58), and
Testing Askarels (D 901 – 56).

Tentative Revision of Standard Specifications for:

Communication and Signal Lime-Glass Insulators (D 879 – 58).

The revisions of Tentative Methods D 150 and D 1458 were accepted by the Standards Committee on December 28, 1959, and the revised methods are available as separate reprints. The tentative revision of Standard Methods D 901 was accepted on September 15, 1959, and the remaining recommendations were accepted on November 23, 1959; they appear in the 1959 Supplement to the Book of ASTM Standards, Part 9.

REPORT OF COMMITTEE D-10
ON
SHIPPING CONTAINERS*

Committee D-10 on Shipping Containers held two meetings during the year: in Madison, Wis., on October 16 and 17, 1958, and in Chicago, Ill., on April 14, 1959.

The committee consists of 90 members, of whom 30 are classified as producers, 35 as consumers, and 25 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On March 25, 1959, the Administrative Committee on Standards, acting for the Society, accepted the following recommendations of Committee D-10:

New Tentative Method of Test for:

Dynamic Properties of Package Cushioning Materials (D 1596 - 59 T).

Revision of Tentative Definitions of:

Terms Relating to Shipping Containers (D 996 - 59 T).

The new and revised tentatives will appear in the 1959 Supplement to Book of ASTM Standards, Part 6.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Method of Vibration Test for Shipping Containers (D 999 - 48 T)[1] and continuation of the method as tentative:

Section 5(*a*).—Revise to read as follows:

5. (*a*) The package shall be placed on the table without fastening. The table shall be operated at a speed that will result in output forces of the table equivalent to 1.0 to 1.1 times gravity (Note).

NOTE.—*Machine Calibration.*—To determine the speed at which a given amplitude produces an acceleration of one times gravity, it is recommended that an accelerometer capable of sensing 2 *g* accelerations at frequencies up to 10 cycles per second (600 cpm) be secured to the table in the area where the package is to be tested and the machine speed then varied until an acceleration of one times gravity is indicated on a suitable indicating or recording device. If tests require accelerations greater or less than one times gravity, it is recommended that the above procedure be employed to determine the proper table speed.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

Drop Test for Shipping Containers (D 775 - 57 T),
Testing Pallets (D 1185 - 51 T),
Test for Water Vapor Permeability of Packages by Cycle Method (D 1251 - 53 T),
Test for Water Vapor Permeability of Shipping Containers by Cycle Method (D 1276 - 53 T), and
Testing Package Cushioning Materials (D 1372 - 55 T).

The recommendations in this report have been submitted to letter ballot of

---

*** Sixty-second Annual Meeting of the Society, June 21-26, 1959.**

[1] 1958 Book of ASTM Standards, Part 6.

the committee, the results of which will be reported at the Annual Meeting.[2]

ADVISORY COMMITTEE ACTION

The Advisory Committee, by action at its meeting on October 16, 1958 and confirmed on April 14, 1959, reorganized the structure of Committee D-10 by the discharge of three subcommittees (II, Methods of Testing; IV, Performance Standards; and V, Correlation of Tests), the elevation of five task groups to the status of subcommittee, and the organization of a new subcommittee on Methods Appraisal.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definition of Terms (C. S. Macnair, chairman) will continue its consideration of terms to be added to Definitions D 996, but will limit its scope to those terms specifically referred to in methods developed and written by Committee D-10.

Subcommittee II on Methods Appraisal (C. R. Gustafson, chairman) is a new group organized to review existing standards and tentatives and to consider proposals for new areas of investigation, with authority to assign priority for Committee D-10 investigation on the basis of need and merit.

Subcommittee III on Moisture and Water Vapor Resistance (H. A. Bergstrom, chairman) has completed revision of the Method of Test for Water Vapor Permeability of Shipping Containers (D 1008). Other permeability methods are scheduled for review.

Subcommittee IV on Drop Test (E. H. Borkenhagen, chairman) has completed revision of Methods D 775 and has been placed on inactive status.

Subcommittee V on Revolving Drum Test (M. J. Clark, chairman) has completed revision of Method of Test for Shipping Containers in Revolving Hexagonal Drum (D 782 – 47).

Subcommittee VI on Interior Packing (K. Q. Kellicutt, chairman) continues its development of a method of test for compatibility of cushioning materials.

Subcommittee VII on Stacking Test (K. R. Martin, chairman) is continuing its development of a method of test for compression under static load.

Subcommittee VIII on Vibration Test (A. M. Bak, chairman) has completed the revision of Method D 999 referred to earlier in the report.

Subcommittee IX on Incline Impact Test (G. B. Clark, chairman) is reviewing the construction of the incline impact testers currently used in the shipping container industry.

This report has been submitted to letter ballot of the committee which consists of 90 members; 85 members returned ballots, of whom 83 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. G. TURK,
Chairman.

R. F. UNCLES,
Secretary.

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the following recommendation:

Revision and Reversion to Tentative of Standard Methods of Test for:

Water Vapor Permeability of Shipping Containers (D 1008 – 51).

This recommendation was accepted by the Standards Committee on December 28, 1959, and the revised tentative methods are available as a separate reprint.

REPORT OF COMMITTEE D-11
ON
RUBBER AND RUBBER-LIKE MATERIALS*

Committee D-11 on Rubber and Rubber-Like Materials and its subcommittees met twice during the year: in Boston, Mass., on June 27, 1958, and in Pittsburgh, Pa., on February 6, 1959. At both meetings, recommendations were made for several new tentatives and revisions of existing standards.

At the Annual Meeting in June, 1958, the present officers were reelected for the ensuing term of two years:

Chairman, S. Collier.

Vice-Chairman, H. G. Bimmerman.

Secretary, J. J. Allen.

It was also voted to increase the Advisory Committee membership from seven to ten. W. A. Frye and I. D. Patterson were elected as new members, and Messrs. A. W. Carpenter, S. Collier, H. G. Bimmerman, L. V. Cooper, G. C. Maassen, R. A. Schatzel, and R. F. Tener were reelected.

Mr. W. C. Tyler was appointed to represent Committee D-11 on the new Committee E-15 on Analysis and Testing of Industrial Chemicals.

The plans for the ISO/TC 45 on Rubber meeting in New York from October 26 through 31, 1959, are well under way. Headquarters will be at the Henry Hudson Hotel. The Plenary Sessions and meetings of the working groups will be held there. A banquet for official foreign and American delegates will be held on Thursday evening, October 29. Committee D-11, through the American Standards Assn., is sponsoring this meeting.

Committee D-11 is cooperating with the American Chemical Society's Rubber Division and The American Society of Mechanical Engineers' Rubber and Plastics Division in sponsoring the International Rubber Conference to be held in Washington, November 8 through 13, 1959. There will be 12 technical sessions and a banquet.

Two technical papers were given at the meeting in February, 1959: "The Langley as a Unit for Timing Outdoor Exposures," by C. R. Caryl,[1] and "Mooney Cure Tests for Calculating Curing Times of Rubber" by A. E. Juve.[2] These papers will appear in the ASTM BULLETIN.

During the past year there have been several losses in membership due to retirement and also the addition of new members. Mr. O. M. Hayden, a former chairman of Committee D-11 (1936 to 1942), has retired from the E. I. du Pont de Nemours Co., Inc. Mr. Hayden was honored at a dinner of Committee D-11 at the Annual Meeting in Boston in June, 1958, and was elected an Honorary Member of the committee.

Financial Status.—Committee D-11 felt the need to raise funds for unusual expenses in connection with its operation. Two requests for voluntary contributions have been made and the results were very gratifying. The financial report to date shows $1779.82 in the D-11 fund.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Published in the ASTM BULLETIN, No. 243, Jan., 1960, p. 55.

[2] Published in the ASTM BULLETIN, No. 239, July, 1959, p. 71.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentatives:

Specifications and Methods of Test for Sponge Made from Closed Cell Poly(Vinyl Chloride) or Copolymers Thereof (D 1667 - 59 T), and

Method of Test for Abrasion Resistance of Rubber Soles and Heels (D 1630 - 59 T).

Revisions of Tentatives:

Methods for Chemical Analysis of Rubber Products (D 297 - 55 T),

Methods of Testing Hard Rubber Products (D 530 - 57 T),

Methods of Test for Indentation of Rubber by Means of a Durometer (D 676 - 58 T),

Description of Types of Styrene-Butadiene Rubbers (D 1419 - 58 T), and

Description of Types of Styrene-Butadiene and Butadiene Rubber Latices (D 1420 - 58 T).

These recommendations, with the exception of Specifications and Method of Test D 1667, were accepted by the Standards Committee on March 25, 1959, for publication by the Society. Specifications and Method D 1667 were accepted on July 14, 1959.

NEW TENTATIVES

The committee recommends for publication as tentative the following specification and method of test as appended hereto:[3]

Tentative Specification for:

Synthetic Rubber Heat and Moisture Resisting Insulation for Wire and Cable, 75 C Operation, developed by Subcommittee V.

Tentative Method of Testing:

Automotive Air Conditioning Hose, developed by the SAE-ASTM Technical Committee on Automotive Rubber.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Sample Preparation for Physical Testing of Rubber Products (D 15 - 58 T):[4]

Table II.—Add the SBR number 1508 to column I-B, and add the SBR number 1603 to column VI-B.

Tentative Specifications for Friction Tape for General Use for Electrical Purposes (D 69 - 57 T):[4]

Section 13.—Add an additional sentence to read, "No individual thickness shall vary from the nominal thickness more than ±0.004 in. and no individual width shall vary more than $\pm\frac{1}{16}$ in."

Section 20.—Insert a new Paragraph (*c*) to read as follows, relettering the present Paragraph (*c*) as Paragraph (*d*): "The oven shall conform to the requirements of Section 4 of Method D 573."

Tentative Specifications for Rubber Insulating Tape (D 119 - 57 T):[4]

Section 8.—Add an additional sentence at the end of the paragraph to read: "No individual thickness shall vary from the nominal thickness more than ±0.004 in. and no individual width shall vary more than $\pm\frac{1}{16}$ in."

Section 20.—Change to read as follows:

20. The thickness of the tape and separator shall be determined by means of a micrometer graduated to 0.001 in. and having a presser foot 0.25 ± 0.01 in. in diameter, exerting a total force of 9 ± 0.1 oz. The load shall be applied by means of a weight. The thickness of the separator shall be determined in a similar manner. The thickness of the separator subtracted from the thickness of the tape and separator shall be considered the thickness of the tape. Five thicknesses and five width measurements shall be made at random in a length of not less

---

[3] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 9.

[4] 1958 Book of ASTM Standards, Part 9.

than 3 ft on each sample roll and the average of these five measurements shall be the average thickness and width referred to in Section 8.

Tentative Methods of Testing Rubber and Thermoplastic Insulated Wire and Cable (D 470 - 58 T):[4]

Section 41(a).—Change to read: "(*a*) The insulation shall show no cracking or surface checking visible to the unaided eye."

Tentative Method of Test for Change in Properties of Elastomeric Vulcanizates Resulting from Immersion in Liquids (D 471 - 57 T):[4]

Table II—Add Reference Fuel C as follows:

Reference Fuel C	*Iso*octane,[a]	50 volumes
	Toluene,[b]	50 volumes

Note 2.—Add the following sentence: "Reference Fuel C has a swelling action on elastomeric vulcanizates more severe than Fuel B."

Tentative Specifications for Elastomer Compounds for Automotive Applications (D 735 - 58 T):[4]

Section 2(c).—Add to the list of suffix letters the following:

S_1.......	Low-Temperature Stiffness at −40 F
S_2.......	Low-Temperature Stiffness at −67 F

Revise suffix letters F_1 and F_2 to read as follows:

F_1.......	Low-Temperature Brittleness at −40 F
F_2.......	Low-Temperature Brittleness at −67 F

Section 5.—Revise Paragraph (*h*) to read as follows:

(*h*) *Low-Temperature Suffixes F_1 and F_2.*—Tentative Method of Test for Brittleness Temperature of Plastics and Elastomers by Impact—Procedure B (ASTM Designation: D 746) (Note).

NOTE.—Some upgrading of quality may be required in some cases because of the slightly greater severity of Method D 746 over the method previously specified, namely, Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (ASTM Designation: D 736).

Add a new Paragraph (*p*) to read as follows:

(*p*) *Low Temperature Stiffness Suffixes S_1 and S_2.*—Tentative Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by Means of a Torsional Wire Apparatus (ASTM Designation: D 1053), Section 9. A Young's modulus of 10,000 psi maximum at the specified temperature is to be the limit of acceptable stiffness.

Table III.—Revise the column headings and the values given for compression set as shown in the accompanying Table I.

TABLE I.—REVISION OF TABLE III, SPECIFICATIONS D 735 - 58 T.

Durometer	40	50	60	70	80	90
BASIC REQUIREMENT						
Compression set after 22 hr at 212 F, max, per cent............	60	55	55	55	55	55
SUFFIX B REQUIREMENT						
Compression set after 22 hr at 212 F, max, per cent............	30	25	25	25	25	25

TABLE II.—REVISION OF TABLE IV, SPECIFICATIONS D 735 - 58 T.

Durometer	30	40	50	60	70	80	90
BASIC REQUIREMENT							
Compression set after 22 hr at 212 F, max, per cent............	80	80	80	80	80	80	80
SUFFIX B REQUIREMENT							
Compression set after 22 hr at 212 F, max, per cent............	35	35	35	35	35	35	35

Table IV.—Revise the column headings and the values given for compression set as shown in the accompanying Table II.

Table V.—Add Grade TA-512 with the following properties:

Basic Requirements:	
Durometer Hardness	50±5
Tensile Strength, psi	1200
Elongation, per cent	450
Heat Aged 70 hr at 437 F:	
Durometer Hardness Change	+15 max
Tensile Strength Change, per cent	−50 max
Elongation Change, per cent	−50 max
Suffix Requirements:	
Suffix B—Compression Set 70 hr at 302 F, per cent	60 max
Suffix E_1—ASTM No. 1 Oil 70 hr at 302 F:	
Tensile Strength Change, per cent	50 max
Elongation Change, per cent	50 max
Durometer Hardness Change, per cent	−15 max
Volume Change, per cent	0 to +20
Suffix E_3—ASTM No. 3 Oil 70 hr at 302 F	non-resistant
Suffix L—Water Absorption after 70 hr boiling:	
Durometer Hardness Change	−10 max
Volume Swell, per cent	+5 max

Tentative Method of Test for Compressibility and Recovery of Gasket Materials (D 1147 – 56 T):[4]

Section 2(c).—Revise to read: "(*c*) *Dial.*—An indicating dial or dials, graduated in 0.001 in. to show the thickness of the specimen during the test."

Section 4.—Letter the present Paragraph as (*a*), and add a new Paragraph (*b*) to read as follows:

> (*b*) If a mechanical means of maintaining 50 to 55 per cent RH is not available, a tray containing a saturated solution of reagent grade magnesium nitrate ($Mg(NO_3)_2 \cdot 6H_2O$) shall be placed in the chamber to provide the required relative humidity. In all cases where testing is conducted outside the area of specified humidity, specimens shall be removed from the chamber one at a time as needed.

Table I.—Revise to read as shown in the accompanying Table III.[5]

Tentative Specifications for Nonmetallic Gasket Materials for General Automotive and Aeronautical Purposes (D 1170 – 58 T):[4]

Table III.—Add Identification No. P3441A with the following values:

Compressibility under 1000 psi total load	5 to 15 per cent	
Recovery under 20 psi	45 min, per cent	
Tensile strength, min, psi	2200	
Properties after immersion in liquids for 22 hr at 70 to 85 F:	Thickness Increase, max, per cent	Weight Increase, max, per cent
ASTM Reference Fuel B	20	30
ASTM Oil No. 3	20	30
Distilled Water	30	30

Tentative Specifications for Ozone Resistant Rubber Insulating Tape (D 1373 – 57 T):[4]

Section 9.—Add the following sentence: "No individual thickness shall vary from the nominal thickness more than ±0.004 in. and no individual width shall vary more than $\pm\frac{1}{16}$ in."

Section 21.—Change to read as recommended above in Section 20 of Method D 119.

Tentative Recommended Practice for Description of Types of Styrene-Butadiene Rubbers (SBR) (D 1419 – 59 T):[4]

Table II.—Add types 1507, 1508, 1603, 1713, and 1714, with the values shown in the accompanying Table IV. Add symbol PAR = Paraffinic.

---

[5] The revised table is not included in this report but appears in the revised method of test; see 1959 Supplement to Book of ASTM Standards, Part 9.

TABLE IV.—ADDITIONS TO TABLE II, RECOMMENDED PRACTICE D 1419 – 58 T.

Type	1507	1508	1603	1713	1714
Nominal Temperature, deg Fahr	43	43	43	43	43
Activator	FRA	FRA	FRA	FRA	FRA
Shortstop	ND	ND	ND	ND	ND
Antioxidant	NST	NST	NST	NST	ST
Catalyst	OHP	OHP	OHP	OHP	OHP
Emulsifier	Mixed	FA	FA	Mixed	Mixed
Nominal Bound Styrene, per cent	23.5	23.5	23.5	23.5	23.5
Conversion, per cent	60	60	60	60	60
Viscosity	35	52	....	52	52
Coagulation	SA	SA	GA	SA	SA
Carbon Black Type	....	....	EPC	....	....
Carbon Black, per cent	....	....	33.4	....	....
Oil Type	....	....	....	NAPH	HI-AR
Oil Parts	....	....	....	50	50
Finishing	normal	normal	normal	normal	normal

Section (*2*).—In Paragraph (*c*) revise the table of producers code number and product type to read as follows:

Producers Code No.	Product Type
50 to 99	Hot Black Masterbatch with 14 or less parts of oil per 100 SBR
150 to 199	Cold Black Masterbatch with 14 or less parts of oil per 100 SBR
250 to 299	Cold Oil Black Masterbatch with 15 or more parts of oil per 100 SBR

Add a new Paragraph (*d*) to read as follows:

(*d*) It is suggested that, when using regular numbers in the ranges listed in Table II, the black masterbatches and black-oil masterbatches be numbered as follows:

1600 to 1699 for Cold Black Masterbatches with 14 or less parts of oil per 100 SBR
1800 to 1899 for Cold Oil Black Masterbatches with 15 or more parts of oil per 100 SBR

Tentative Recommended Practice for Description of Types of Styrene-Butadiene Rubber (SBR) and Butadiene Rubber (BR) Latices (D 1420 – 59 T):[6]

Table I.—Add types 2109 and 2113, with the following properties:

Type	2109	2113
Nominal Temperature, deg Fahr	50	43
Activator	FRA	FRA
Shortstop	ND	ND
Catalyst	OHP	OHP
Emulsifier	FA	Mixed
Nominal Conversion, per cent	60	80
Contained Polymer Nominal Mooney Viscosity, ML 1 + 4(212 F)	...	140
Nominal Residual Volatile Unsaturate, per cent	0.1	0.10
Nominal pH Value	10.5	10.5
Nominal Surface Tension, dynes per cm	...	...
Nominal Coagulum on No. 80 Screen, per cent	0.10	...
Nominal Bound Styrene, per cent	40	44
Nominal Total Solids, per cent	39.5	48

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Methods of Test for Abrasion Resistance of Rubber Compounds (D 394 – 47):[4]

Revise as appended hereto.[7] This

---

[6] 1959 Supplement to Book of ASTM Standards, Part 9.

[7] The revised method of test appears in the 1959 Supplement to Book of ASTM Standards, Part 9.

method has been completely rewritten and eliminates Procedure B which is now part of the new Tentative Method of Test for Abrasion Resistance of Rubber Soles and Heels (D 1630 – 59 T). The revised method also cross references the standard formulations to Methods D 15.

Standard Methods of Test for Mechanical Properties of Elastomeric Vulcanizates Under Compressive or Shear Strains by the Mechanical Oscillograph (D 945 – 55):[4]

Section 2(b).—Since V-Mail scrip ink is no longer available, change the first sentence of Note 3 to read: "A suitable black ink may be used in the pen in order to obtain records that can be blueprinted."

Figures 5 and 6.—Change letter *D* to *P* and letter *E* to *Q*. Add letters *D* and *E* to the next oscillogram reversals following *A*, *B* and *C*.

Section 9.—Change Paragraph (*d*) to read:

(*d*) *Yerzley Resilience in per cent* shall be determined as the average computed from the second and third half cycles:

$$\text{Yerzley resilience, per cent} = \left\{\frac{CD}{BC} + \frac{DE}{CD}\right\} \times 50$$

In Paragraph (*i*) change the letters *DE* to *PQ*.

New Note.—Add a new Note 5 to read:

NOTE 5.—This method of computing resilience may produce results which are 3 to 4 percentage points higher than the results as formerly computed by the previous use of *BC* divided by *AB* times 100.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Rubber Insulating Blankets (Without Fabric Reinforcement) (D 1048 – 49 T),[4]

Rubber Insulator Hoods (D 1049 – 49 T),[4]

Rubber Insulating Line Hose (D 1050 – 49 T),[4]

Concentrated, Ammonia Preserved, Creamed and Centrifuged Natural Rubber Latex (D 1076 – 57 T),[4]

Nonrigid Thermoplastic Compounds for Automotive and Aeronautical Applications (D 1277–53 T),[4] and

Sheet Rubber Packing (D 1330 – 55 T).[4]

Tentative Methods of:

Testing Rubber Hose (D 380 – 57 T),[4]

Dynamic Testing for Ply Separation and Cracking of Rubber Products (D 430 – 57 T),[4]

Testing Compressed Asbestos Sheet Packing (D 733 – 57 T),[4]

Test for Resistance of Vulcanized Rubber or Synthetic Elastomers to Crack Growth (D 813 – 57 T),[4]

Conditioning of Elastomeric Materials for Low-Temperature Testing (D 832 – 56 T),[4]

Test for Weather Resistance Exposure of Automotive Rubber Compounds (D 1171 – 57 T),[4] and

Test for Penetration of Hard Rubber by Type D Durometer (D 1484 – 57 T).[4]

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Specifications for Rubber Insulating Sleeves (D 1051 – 56 T):[4]

Section 3(a).—Revise the title to read: "Voltage Test and Proof Test Current."

Section 6.—Revise to read: "The sleeves shall be free from harmful physical defects which can be detected by thorough test or inspection."

Tentative Methods for Testing Synthetic Rubber Latices (Styrene-Butadiene Copolymers) (D 1417 – 57 T):[4]

Table I.—Add types 2076, 2107, 2108, 2109, 2110, 2111, 2112, and 2113, with a drying aid of 1 ml of distilled water and a drying time of 45 min.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the remaining tentatives under its jurisdiction. Many of them are under consideration for revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[8]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Rubber Hose (R. A. Bonnell, chairman) completed a review of the Tentative Methods of Testing Rubber Hose (D 380 – 57 T) and has recommended that they be adopted as standard.

Subcommittee II on Belting (F. M. Galloway, chairman) is making a review of the Tentative Methods of Testing Flat Rubber Belting (D 378 – 51 T). Mr. Galloway has found it necessary to resign as chairman of this subcommittee and recommended that Earl Webster of the same company, Quaker Division of H. K. Porter Co., be appointed chairman. This action will be approved at the Annual Meeting.

Subcommittee III on Tests of Rubber Thread (K. F. Cullison, chairman) is reviewing and conducting tests on the Proposed Methods of Testing Rubber Thread which have been published as information since 1950. It hopes to submit these methods after revision as tentative.

Subcommittee IV on Protective Equipment for Electrical Workers (Gordon Thompson, chairman) has recommended that the several tentatives under its jurisdiction be adopted as standard. The specifications for gloves are under revision.[9]

Subcommittee V on Insulated Wire and Cable (J. T. Blake, chairman) has recommended another tentative specification and is working on revisions of the Tentative Specification for Construction of Rubber Insulated Wire and Cable (D 1350 – 58 T).

Subcommittee VI on Packings (R. F. Anderson, chairman) is continuing its work on revisions of Methods D 1147. It is also working with Section X-C on Gaskets of the SAE-ASTM Technical Committee on Automotive Rubber on revisions of Specifications D 1170.

Subcommittee VII on Rubber Latices (G. H. Barnes, chairman) has recommended that the Specifications and Methods of Test for Concentrated, Ammonia Preserved, Creamed and Centrifuged Natural Latex (D 1076 – 57 T) be adopted as standard.

Subcommittee VIII on Nomenclature and Definitions (R. G. Seaman, chairman) has the difficult problem of developing acceptable definitions for the terms "rubber" and "rubber-like." With the many new elastomeric materials now in use and to be developed, the need for revised definition is quite urgent. It is asking for help and suggestions.

Subcommittee IX on Insulating Tape (C. W. Pickells, chairman) has recommended revisions of the tentatives under its jurisdiction. It is working on a tentative specification for water-resisting rubber tape.

Subcommittee X on Physical Testing of Rubber Products (L. V. Cooper, chairman) has reactivated a Task Group on Testing of Rubber O-Rings under the chairmanship of D. S. Messenger.

Subcommittee XI on Chemical Analysis of Rubber Products (W. P. Tyler, chairman) is considering the possibility of consolidating and clarifying methods of analysis in the present Methods D 297, D 833, and D 1278.

---

[8] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[9] See Editorial Note, p. 487.

Subcommittee XII on Crude Natural Rubber (Norman Bekkedahl, chairman) is working with Subcommittees X and XXIX on physical testing methods for evaluation of crude rubber.

Subcommittee XIII on Synthetic Elastomers (B. S. Garvey, Jr., chairman) has the following work in progress: sampling procedures for SBR-black masterbatches; chemical methods for determining oil content of SBR-oil masterbatches; and establishment of a formula for testing SBR-oil masterbatches.

Subcommittee XIV on Abrasion Tests for Rubber Products (R. F. Tener, chairman) has revised the methods for abrasion testing (D 394). A new method for testing the abrasion resistance of heels and soles has also been recommended.

Subcommittee XV on Life Tests for Rubber Products (G. C. Maassen, chairman) is making progress on the development of an oven and method for oven aging of highly plasticized elastomers and plasticized PVC. This is a joint effort with Subcommittee V and with Committee D-20 on Plastics. A new problem involving a method for the determination of staining of white compounds when in contact with compounds containing carbon black is under way.

Subcommittee XVI on Classification and Specifications of Rubber Compounds (J. F. Kerscher, chairman) is working with ISO/TC 45 on Rubber and Section IV-D of the Technical Committee on Automotive Rubber on a revised method for the classification of rubber compounds. Progress is being made.

Subcommittee XVII on Tests for Hardness, Set, and Creep (S. R. Doner, chairman) has under way a review of the present compression set method.

Subcommittee XVIII on Flexing Tests (Howard Tangenberg, chairman) has recommended that Tentative Methods D 430 and D 813 be adopted as standard.

Subcommittee XIX on Tests for Properties of Rubber and Rubber-Like Materials in Liquids (F. H. Fritz, chairman) is considering revisions of Method D 471 anticipating future requirements for new fluids, higher temperatures, and improvements in accuracy of measurements.

Subcommittee XX on Adhesion Tests (H. H. Irvin, chairman) is cooperating with Subcommittee XXI on problems of adhesion pertaining to textiles, wire, and also cured rubber to metal. They are considering revisions of Methods D 429 to include environmental exposure aspects. They are also studying the measurement of rubber to metal adhesion by nondestructive methods.

Subcommittee XXI on Testing of Rubber Cements and Related Products (J. F. Anderson, chairman) reports that a new task group has been formed under the chairmanship of A. E. Hicks to develop methods of test for tire cord adhesion. This will include both textile cord and wire cord.

Subcommittee XXII on Cellular Rubber (H. G. Bimmerman, chairman) is cooperating with the Society of Plastics Industries, Committee D-20 on Plastics, the Rubber Manufacturers Assn., and the Technical Committee on Automotive Rubber on the revisions of specifications and methods of test for urethane and poly(vinyl chloride) foam and sponge products.

Subcommittee XXIII on Hard Rubber (W. J. Dermody, chairman) has under consideration revisions of Methods D 530, including impact methods and a possible tabulation of hard rubber grades. Consideration is also being given to the expansion of Methods of Testing Asphalt Composition Battery Containers (D 639) to include hard rubber battery containers.

Subcommitte XXIV on Rubber and Rubber-like Coated Fabrics (K. L. Keene, chairman) is making progress on methods for flame resistance, cold resistance, and tack-tear test of coated fabrics.

Subcommittee XXV on Low-Temperature Tests (R. S. Havenhill, chairman) has set up a round-robin test on a proposed method for measuring the speed of the Scott solenoid low-temperature brittleness tester.

Subcommittee XXVI on Processibility (R. H. Tayler, chairman) is studying the possibility of combining Methods D 927 and D 1077 into one standard.[9] It is investigating the use of the extrusion methods of measuring plasticity. A task group is studying the problem of measuring shrinkage of raw and compounded polymers.

Subcommittee XXVII on Tests of Resilience (W. A. Frye, chairman) has recommended revisions in the method of measuring resilience with the Yerzley oscillograph (D 945). A general method of forced vibration testing is under consideration.

Subcommittee XXVIII on Statistical Quality Control (E. M. Bader, chairman) continues to assist other subcommittees in setting up test programs.

Subcommittee XXIX on Compounding Ingredients (A. E. Juve, chairman) has as its main objective the rewriting of Methods D 15, Physical Tests for Rubber Products, and its combination with Methods D 1522, Testing Carbon Blacks in Rubber. This involves formulations for standard compounds and mixing procedures.

The SAE-ASTM Technical Committee on Automotive Rubber (W. J. Simpson, chairman) is actively engaged in the preparation of a new format for classifying rubber compounds. A new Section XV has been organized to prepare specifications and test methods for suspension bushings. Section X has been reorganized. Mr. M. H. Kapps is chairman of a new Section X on Gaskets; Mr. R. C. Waters is chairman of Section XI which includes Subsection A on O-Rings and Subsection B on Oil Seals.

This report has been submitted to letter ballot of the committee, which consists of 257 members; 186 members returned their ballots, of whom 174 have voted affirmatively and 3 negatively.

Respectfully submitted on behalf of the committee,

S. COLLIER,
Chairman.

JOHN J. ALLEN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Low-Voltage Insulating Gloves (D 1700 – 59 T).

New Tentative Method of Test for:

Viscosity and Curing Characteristics of Rubber by the Shearing Disk Viscometer (D 1646 – 59 T), which is a revision and combination of Methods D 927 and D 1077.

Revision of Tentative Specifications for:

Rubber Insulating Gloves (D 120 – 52 T), and
Ozone Resisting Butyl Rubber Insulation for Wire and Cable (D 1352 – 54 T).

Revision of Tentative Methods of:

Sample Preparation for Physical Testing of Rubber Products (D 15 – 59 T), and
Testing Rubber Coated Fabrics (D 751 – 57 T).

Revision of Tentative Recommended Practice for:

Description of Types of Styrene-Butadiene Rubbers (D 1419 – 59 T).

Revision of Tentative Specifications and Methods of Test for:

Latex Foam Rubbers (D 1055 – 58 T),
Flexible Urethane Foam (D 1564 – 58 T),
Flexible Foams Made from Polymers or Copolymers of Vinyl Chloride (D 1565 – 58 T), and
Sponge and Expanded Cellular Rubber Products (D 1056 – 58 T).

Revision and Reversion to Tentative of Standard Specifications for:

Ozone Resisting Insulation for Wire and Cable (D 574 – 58).

Revision and Reversion to Tentative of Standard Methods of Testing:

Adhesives for Brake Lining and Other Friction Materials (D 1205 – 58).

Withdrawal of Tentative Method of Test for:

Viscosity of Rubber and Rubber-like Materials by the Shearing Disk Viscometer (D 927 – 57 T), and
Cure Characteristics of Vulcanizable Rubber Mixtures During Heating by the Shearing Disk Viscometer (D 1077 – 55 T).

These recommendations were accepted by the Standards Committee on November 23, 1959, with the exception of the revision of Tentative Specifications D 1352 which was accepted on December 28, 1959. The new and revised tentatives appear in the 1959 Supplement to the Book of ASTM Standards, Part 9.

REPORT OF COMMITTEE D-12
ON
SOAPS AND OTHER DETERGENTS*

Committee D-12 on Soaps and Other Detergents held one meeting during the year: on March 9 and 10, 1959, in New York City. Two papers were presented at the meeting: "Adsorption of Labeled Sodium Carboxymethyl Cellulose by Textile Fibers in Relation to Detergency Action," by J. W. Hensley, Wyandotte Chemicals Corp.; and "A Laboratory Dishwashing Test for Household Detergents," by W. G. Spangler, Colgate-Palmolive Co.

The Committee D-12 Award for outstanding performance in the field of soaps and detergents was presented to Chairman Jay C. Harris by A. M. Schwartz in recognition and appreciation of the outstanding contributions and service in the field of detergents and detergency, and for inspirational leadership. Mr. Harris was secretary of the committee from 1946 to 1950, and has served as chairman since that date.

The committee consists of 119 members, of whom 57 are classified as producers, 34 as consumers, and 28 as general interest members, with 4 consulting members.

Accepted with deep regret was the resignation of Vice-Chairman W. H. Koch, who was also chairman of Subcommittee T-4. His forceful promotion of committee work will be sorely missed. E. W. Blank was appointed to replace him as committee vice-chairman, and M. V. Trexler was appointed as chairman of Subcommittee T-4. Resignation of L. T. Howells, chairman of Subcommittee G-2, Nomenclature and Definitions, led to the appointment of A. M. Schwartz as chairman. D. Price was appointed chairman of Subcommittee T-6 on Metal Cleaning, replacing H. A. Kafarski who resigned.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the proposed Method for Rapid Determination of Solids by Infrared Moisture Balance as appended hereto.[1]

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Synthetic Anionic Active Ingredient in Detergents by Cationic Titration Procedure as appended hereto.[2]

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods for Sampling and Analysis of Sodium Triphosphate (D 501 - 58 T):[3]

Revise by adding as the preferred

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

[1] This proposed method has not been preprinted; copies are available on request at ASTM Headquarters.

[2] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 10.

[3] 1958 Book of ASTM Standards, Part 10.

method the Pressurized Reverse-Flow Ion-Exchange Chromatographic Procedure, as appended hereto,[4] with present Sections 21 to 28 to be retained as an alternative test method.

Tentative Methods of Testing Detergent Alkylate (D 1569 - 58 T):[3]

Revise by adding a new method for acid wash color, as appended hereto.[4]

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following standards:

Standard Specifications for:

Palm Oil Solid Soap (D 535 - 52),
Palm Oil Chip Soap (D 536 - 52), and
Olive Oil Solid Soap (D 592 - 42).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 - 58):[3]

Section 16.—Change the last sentence of the Note to read: "Free alkali values in soap or surfactant mixtures containing borax are unreliable due to solubility of borax in hot alcohol."

Section 52.—Change the last sentence to read: "Larger amounts must be dehydrated but need not be removed by filtration during preparation of the sample."

Standard Methods of Sampling and Chemical Analysis of Alkaline Detergents (D 501 - 58):[3]

Section 20.—Replace the first two sentences with the following:

20. Transfer approximately 1.2 g of sample into a tared weighing bottle. Weigh to the nearest 0.1 mg, protecting the sample at all times, as much as possible, from moisture in the air during weighing. Dissolve the sample in about 50 ml of water in a 400-ml beaker and add 2 drops of methyl orange indicator solution.

Standard Methods of Chemical Analysis of Industrial Metal Cleaning Compositions (D 800 - 58):[3]

Section 26.—Revise the last sentence to read: "Larger amounts must be dehydrated but need not be removed by filtration during preparation of the sample."

Standard Methods of Chemical Analysis of Soaps Containing Synthetic Detergents (D 820 - 58):[3]

Section 7.—Add a new Note to read as follows:

NOTE.—In the analysis of soaps known to contain little or no alkaline salts, it is unnecessary to filter the hot alcoholic soap solution. However, the filtration should be carried out in all cases where alkaline salts such as silicates, phosphates, borates, and similar salts are present, since these are known to affect the free alkali determination. Free alkali figures in soap or surfactant mixtures containing borax are unreliable due to solubility of borax in hot alcohol.

Section 22.—Revise the last sentence to read: "Larger amounts must be dehydrated but need not be removed by filtration during preparation of the sample."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee S-2 on Specifications for Soaps and Synthetic Detergents (W. H. Joy, chairman). — Specifications for liquid scrub soaps, liquid soaps, and various forms of generally accepted industrial cleaning compositions are under consideration.

---

[4] The new method is included in the revised tentative which appears in the 1959 Supplement to Book of ASTM Standards, Part 10.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Task Group 1 on Synthetic Detergents (R. L. Liss, chairman).—Lack of agreement on test methods hindered presentation of a specification for a built, all-purpose, high-sudsing detergent. The available data may suggest more limited form of specification.

Subcommittee T-2 on Analysis of Soaps and Synthetic Detergents (J. C. Harris, chairman):

Task Group 8 on Determination of Sodium Carboxymethyl Cellulose (J. L. Darragh, chairman) expects to recommend a procedure for analysis at the next Annual Meeting.

Task Group 14 on Rapid Determination of Moisture by Infrared Lamp (O. L. Sherburne, chairman) recommended the Method for Rapid Determination of Solids by Infrared Moisture for publication as information only, as noted earlier in the report.

Task Group 15 on Determination of Anionic Active Ingredient in Synthetic Detergents by Cationic Titration (L. E. Weeks, chairman) presented the Tentative Method of Test for Synthetic Anionic Active Ingredients in Detergents by Cationic Titration Procedure referred to earlier in the report.

Task Group 20 on Total Active Ingredient (C. A. Cohen, chairman) has a revised procedure in preparation.

Task Group 21 on Detergent Alkylate (J. C. Harris, chairman):

Sub-Group A on Distillation Range (J. L. Darragh, chairman) is evaluating a procedure based on D-2 standards.

Sub-Group C on Acid Wash Color (R. C. Stillman, chairman) recommended the revision of Methods D 1569 referred to earlier in the report.

Sub-Group D on Standard Sulfonation Procedure and Analysis of Product (C. A. Cohen, chairman).—A more satisfactory flask has been designed and will be tested before the next Annual Meeting.

Subcommittee T-4 on Analysis of Inorganic Alkaline Detergents (M. V. Trexler, chairman).—A task group headed by C. H. Russell presented cooperative evaluation data for the Pressurized Reverse Flow Ion-Exchange Chromatographic Analysis of Sodium Tripolyphosphate used in the revision of Methods D 501 referred to earlier in the report.

Subcommittee T-5 on Physical Testing (M. G. Kramer, chairman):

Task Group 3 on Measurement of Reflectance of Test Fabric (R. L. Liss, chairman) analyzed the results of cooperative tests showing need for improvement in technique and expects to have a tentative procedure for the next Annual Meeting.

Task Group 8 on Methods for Evaluation of Rug Cleaning Detergents (J. W. Rice, chairman) reported the development of a standard floor surfacing.

Respectfully submitted on behalf of the committee,

J. C. HARRIS,
Chairman.

H. R. SUTER,
Secretary.

REPORT OF COMMITTEE D-13
ON
TEXTILE MATERIALS*

Committee D-13 on Textile Materials held two general meetings during the year: on October 14 to 17, 1958, and on March 17 to 20, 1959, both in New York, N. Y.

At the October, 1958, session, in addition to meetings of subcommittees and task groups, a Papers Session on the subject of fibers was held under the auspices of Subcommittee A-3 on Wool Fibers.

During the four-day March meeting, the Harold DeWitt Smith Memorial Medal for 1959 was presented to Walter J. Hamburger of the Fabric Research Laboratories. This was the tenth annual award of the Smith Medal and was given to Dr. Hamburger in recognition of his outstanding achievements in promoting the application of engineering concepts to the study of textile fibers and structures as originally proposed by Harold DeWitt Smith. The citation on the scroll presented to Dr. Hamburger reads as follows:

> **To Walter Julian Hamburger as a testimonial in recognition of his outstanding achievements in pioneering the application of engineering concepts to the study of textile fibers and structures as originally proposed by Harold DeWitt Smith; in advancing the fundamental knowledge of fiber-to-fabric relationships; in contributing to the military services, extending the usefulness of textiles under extreme conditions of service; in improving the techniques of textile processing; and in his untiring effort in the interest of textile education.**

Two meetings were held with representatives of the American Association of Textile Chemists and Colorists concerning the minimizing of duplication of test methods and standards by the two organizations. This cooperation has resulted in the elimination of considerable unnecessary effort. The committee has continued its participation in the work of ISO/TC 38 on Textiles and its cooperation with the International Bureau of Standardization of Man-Made Fibers.

Several new liaison appointments with other technical committees and other textile societies were made during the year. Mr. Cameron Baker succeeded Mr. Julian Jacobs, resigned, as chairman of Subcommittee C-4, Publicity.

Work has progressed during the year on the survey of test methods and standards sponsored by Committee D-13 and several revisions have been made. The revisions have been directed toward making the standards more technically correct and easier to understand.

Mr. A. M. Tenney was elected Honorary Member of the committee in recognition of his long years of service and his work in organizing the subcommittees relating to man-made fibers.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 4 new tentatives, the revision of 10 tentatives, the adoption as standard of 42 tentative definitions appearing in ASTM Definitions D 123, the adoption

---

*** Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.**

of 8 tentatives as standard without revision and the adoption of one tentative with revision, the tentative revision of 1 standard, the adoption as standard of a tentative revision of 3 standards, and the immediate adoption of revisions in 4 standards.

The standards and tentatives affected, together with the revisions recommended, are listed in detail in the Appendix.[1]

All other standards and tentatives not specifically referred to are being studied by the respective subcommittees and it is recommended that they be continued in their present status.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 387 voting members; 185 members returned ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

B. L. WHITTIER,
Chairman.

H. A. EHRMAN,
Secretary.

---

[1] See p. 494.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials. These standards and tentatives appear in their present form in the 1958 Book of ASTM Standards, Part 10.

New Tentatives

The committee recommends for publication as tentative the following methods and recommended practice as appended hereto:[1]

Method of Test for:

Construction Characteristics of Woven Fabrics, to replace eventually Sections 3 and 4 and 6 to 8 of the Standard General Methods of Testing Woven Fabrics (D 39 - 49),

Breaking Load and Elongation of Textile Fabrics, to replace eventually Sections 9 to 14 of Methods D 39, and

Seam Breaking Strength of Woven Textile Fabrics.

Recommended Practice for:

Lighting Cotton Classing Rooms for Color Grading.

Revision of Tentatives

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Identification of Fibers in Textiles (D 276 - 57 T):

Section 1.—Add in the proper alphabetical order the names "Polyethylene" and "Vinyon HH."

Section 8 (d).—Add in the proper alphabetical order the names "Polyethylene" and "Vinyon HH."

Table V.—Revise as shown in the accompanying Table I.[2]

Table VI.—Revise as shown in the accompanying Table II.

Tentative Methods of Quantitative Analysis of Textiles (D 629 - 57 T): Revise as appended hereto.[1]

Tentative Methods of Testing and Tolerances for Tire Cords from Man-Made Fibers (D 885 - 58 T): Revise as appended hereto.[1]

Tentative Method of Test for Twist in Single Spun Yarns (Untwist-Twist Method) (D 1422 - 58 T):

Section 4 (a).—Change to read as follows:

4. (*a*) *Twist Tester*, conforming to the requirements given in Section 4 (*a*) of the Method of Test for Twist in Yarns (Direct-Counting Method) (ASTM Designation: D 1423). In addition, the tester shall be provided with a variable tensioning device so constructed that a specific tension may be applied to the specimen only at the start and finish of the test and removed completely during the intervening untwisting and twisting operations.

Note 1.—There are two general types of tension devices in which (*1*) the tension is applied manually and controlled by the displacement of a pendulum from its vertical position (Section 8 (*a*), Item (*1*)), and (*2*) the tension is applied manually by the means of a known dead-weight load (Section 8 (*a*), Item (*2*)).

---

[1] The new and revised tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 10.

[2] The new table is not included in this report but appears in the revised method, see 1959 Supplement to Book of ASTM Standards, Part 10.

Tentative Method of Test for Twist in Yarns (Direct-Counting Method) (D 1423 - 58 T):

Section 2 (a).—Delete the following references to twist definitions: *Twist,* Cable, Twist, Direction of; and Twist, Hawser.

Section 2.—Add the following new Paragraphs (*c*), (*d*), and (*e*), renumbering the subsequent paragraphs accordingly:

(*c*) *Twist, Cable, n.*—A thread, cord, or rope construction in which each successive twist is in the opposite direction to the preceding twist, an S/Z/S or Z/S/Z construction.

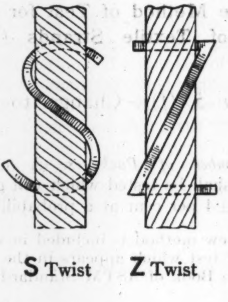
TABLE II.—A SUGGESTED METHOD OF REAGENT APPLICATION FOR FIBER SEPARATION.

NOTE.—Reagents are those shown in Table V.

(Revision of Table VI, Method D 276)

Reagent	Fiber Removed
Acetone, 80 per cent	acetate
Acetone, 100 per cent	Dynel (old)
Acetone, 100 per cent, boil	Dynel (new)
Formic acid (1:1)	nylon 6
Hydrochloric acid	nylon 66
Sulfuric acid	Cuprammonium rayon, silk, viscose, Arnel
Sodium hypochlorite	chlorinated wool, Merinova
Nitric acid	Acrilan, Darvan (partial), Orlon, Zefran
Methylene chloride	Fibravyl, Rhovyl (apparent solubility)
Sodium hydroxide, 45 per cent	Dacron, Saran (melts), Terylene, Vicara

(*d*) *Twist, Direction of, n.*—A yarn has **S** twist if, when held in a vertical position, the spirals around its central axis conform in direction of slope to the central portion of the letter "S," and **Z** twist if the spirals conform in direction of slope with the central portion of the letter "Z."



NOTE.—This definition applies to the twist in single yarns, the ply twist in plied yarns, and the cable or hawser twist in cords.

(*e*) *Twist, Hawser, n.*—A thread, cord, or rope construction in which the single and first ply twist are in the same direction, and the second ply twist is in the opposite direction, an S/S/Z or Z/Z/S construction.

Section 4 (a).—Revise the first four sentences to read as follows, leaving the table and the remainder of the paragraph unchanged:

4. (*a*) Twist counter, consisting of a pair of clamps, one of which is rotatable in either direction and positively connected to a revolution counter. It may be hand or power driven. The position of the one clamp (or both clamps) shall be adjustable to accommodate the specified length of test specimens (Section 8) and to permit measuring the extension or contraction during untwisting. Means shall be provided for pretensioning the specimen, prior to clamping, and for determining the specimen length with an accuracy of ±0.02 in. (0.5 mm). The movable but nonrotatable clamp shall be capable of being traversed with substantially no friction to permit determining the length of the untwisted specimens under the initial tension. The counting device shall be resettable and capable of recording the number of revolutions of the rotatable clamp to the accuracy specified, as follows:

Section 11 (b).—Revise to read as follows, leaving Note 5 unchanged:

(*b*) Mount the specimen in the clamps under a pretension equivalent to 0.25 ± 0.05 g per tex (that is, approximately the weight of 250 m or 273 yd of yarn) (Note 5). Avoid any change in the twist while handling the yarn.

Tentative Method of Test for Number of Neps in Cotton Fibers (D 1446 – 53 T): Revise as appended hereto.[1]

Tentative Method of Test for Dimensional Change of Knit Fabrics (D 1470 – 57 T):

Section 9.—Change to read as follows:

9. (*a*) *Class 3 and Class 2.*—Remove the specimens from the automatic washer after the damp-dry cycle or from the extractor. Dry in a tumble drier at full load, at between 120 and 160 F (49 to 71 C), until dry.

(*b*) *Class 1.*—After the hand-washed specimens have been extracted, lay each on a flat screen without distorting the fabric and dry at room temperature.

Tentative Methods of Test for Pilling Propensity of Textile Fabrics (D 1375 – 55 T):

Title.—Change the word "Propensity" to read "Resistance."

Introduction—In the first line change "propensity" to read "resistance." In the twenty-second and twenty-fourth lines add "resistance" after "pilling."

Section 1.—In the first line change "four" to read "five" and add the following method "E. Random Tumble Pilling Tester."

In the third line, change the word "propensity" to read "resistance."

Section 2.—Add as Paragraph (*e*) the following:

(*e*) *Pilling Resistance.*—Resistance to the formation of pills on a fabric.

Section 17 (*e*).—Change to read as follows:

(*e*) *Optional.*—Standard rating charts for the types of fabrics to be tested, such as tropicals, shirtings, gabardines, worsted flannels, etc.

NOTE 8.—The standards should be stored and handled under conditions that will preserve their original form and appearance. Mounting with thick cardboard framing around the specimens is recommended.

Figures 4 and 5.—Delete and renumber subsequent figures accordingly.

Section 20.—Change to read as follows:

20. (*a*) *Evaluation of Test Specimens.*—Subjectively rate the face of each specimen (as indicated by markings) using the following scale:

5—excellent resistance (no pilling)
4—good resistance (slight pilling)
3—medium resistance (moderate pilling)
2—poor resistance (heavy pilling)
1—very poor resistance (very severe pilling)

If a rating for a test specimen falls between two ratings, assign the half value; that is, 3.5, 2.5, etc.

NOTE 10.—Rating standards made from pilled specimens of the types of fabrics tested, representating the level of pilling equivalent to each of the five rating steps, are valuable as a reference to insure uniform ratings. It is suggested that individual laboratories establish standards based on the ratings for fabrics of particular interest.

(*b*) Check the pilled specimen for nonuniformity of pilling. If the pills are concentrated in any one strip, warpwise or fillingwise, or in any one portion, report this condition. It indicates a variation in the yarn used to prepare the specimen.

(*c*) A correlation should be established through appropriate wear tests for each type of fabric, if absolute, rather than relative, performance is the objective of the test.

Section 21.—Change to read as follows: "21. Report the ratings of all the individual specimens and the average of the three specimens."

New Method.—Add a new Method E. Random Tumble Pilling Tester, as appended hereto.[3]

Tentative Method of Test for Unevenness of Textile Strands (D 1425 – 56 T):

Section 5 (*f*).—Change to read as follows:

(*f*) *Number of Packages.*—A number of packages shall be tested which will give a precision of ±4 per cent at a probability level of

---

[3] The new method is included in the revised method of test which appears in the 1959 Supplement to Book of ASTM Standards, Part 10.

90 per cent. The probability then will be 95 per cent that the true value of unevenness (as would be determined by testing an infinite number of packages) will be no more than 4 per cent above the mean of the test results. The required number of packages will depend upon the variation in unevenness from package to package. If the amount of this variation is not known, then 10 packages shall be tested. If it is known, then the number of packages is equal to 0.17 × (coefficient of variation of the unevenness values)^2 and the following table can be used:

Coefficient of Variation of the Unevenness Values (see Note), per cent	Number of Packages to be Tested
3	2
4	3
5	5
6	6
7	9
7.5	10
8	11
9	14
10	17
11	21
12	25

In the ninth line of the note that follows the table, change "12 packages" to read "10 packages."

Section 5 (g).—In the last line, change "7 per cent" to read "7.5 per cent."

Section 5 (h).—In the fourth line, change "95 per cent" to read "90 per cent."

Tentative Method of Test for Breaking Strength of Yarn by Skein Method (D 1578 – 58 T):

Section 8.—Change to read as follows:

8. (*a*) Unless otherwise agreed upon (for instance, provided for by an applicable materials specification), the number of test specimens shall be such that the mean of the test results will, with 95 per cent probability, be no more than 4 per cent above the true average skein strength (as would be determined by an infinite number of tests). This is equivalent to a precision of ±4 per cent at a probability level of 90 per cent.

$$n = 0.17\, v^2$$

where:

n = number of test specimens, and

v = coefficient of variation of individual test results determined from extensive past records on similar material.

(*b*) If v is not known, perform 10 tests on cotton-system yarns and worsted-spun yarns and 20 tests on woolen-spun yarns.

NOTE 3.—These numbers of tests are based on v = 7.5 per cent for yarns spun on the cotton and worsted systems and on v = 11.0 per cent for woolen-spun yarns. These are somewhat higher values of v than will usually be found in practice. Knowledge of the v actually applying is therefore likely to permit making fewer tests than prescribed in this section.

(*c*) The required number of test specimens shall be distributed as evenly as possible over the lot to be tested. In any case, test a minimum of five ends.

TENTATIVE REVISION OF STANDARD

The committee recommends as a tentative revision of the Standard Definitions of Terms Relating to Textile Materials (D 123 – 58) the following two new tentative definitions to read as follows:

Cashmere Hair, Capra hircus (goat).—The fibers produced by a form of goat (*Capra hircus*) indigenous to Asia and known as the Cashmere goat; characteristically, Cashmere Goat Hair consists of fine down (undercoat) fibers and coarse (outercoat) fibers.

Cashmere.—The down (undercoat) fibers derived from Cashmere Goat Hair and having a diameter of 30 microns or less.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Man-Made Staple Fibers (D 540 – 57 T),

Relaxation and Felting Shrinkage in Laundering of Stabilized Knit Wool Fabrics (D 1284 – 53 T), and

Tear Resistance of Woven Fabrics by Falling-Pendulum (Elmendorf) Apparatus (D 1424 – 56 T).

Tentative Specifications and Method of Test for:

Fineness of Mohair Tops (D 1381 – 57 T).

Adoption of Tentative as Standard with Revisions

The committee recommends that the Tentative Method of Test for Non-Lint Content of Cotton (Shirley Analyser Method) (**D 1451 – 56 T**) be approved for reference to letter ballot of the Society for adoption as standard with the following revisions:

Section 9.—In Paragraph (*a*) change "four" to read "three" and change Paragraph (*b*) to read as follows:

> (*b*) *Ginning or Processing Waste.*—Unless otherwise specified, one test shall be made using the whole sample (taken as specified in Section 5 (*b*)).

Adoption of Tentative Revision as Standard

The committee recommends that the definitions for the following 42 terms now published as tentative revisions of the Standard Definitions of Terms Relating to Textile Materials (**D 123 – 58**) be approved for submission to letter ballot of the Society for adoption as standard, without revision:

Bleaching
Bright
Compliance
Conformance
Creep
Creep, Primary
Creep, Secondary
Curve, Load-Deformation
Curve, Stress-Strain
Curve, Tensile Hysteresis
Deformation
Deformation, Delayed; Creep
Deformation, Immediate Elastic
Dull
Fiber, Man-Made
Fiber, Natural
Gaiting, *n. Warp Knitting*
Grade, *n. Warp Knitting*
Inches per Rack (IPR), Warp Knitting
Luster
Modulus, *Textile, n.*
Modulus, Secant, *n.*
Modulus, Tangent, *n.*
Modulus, Young's
Rack, *n. Warp Knitting*
Ribbon, Slit or Cut
Ribbon, Woven
Staple
Strain, Tensile
Strength, Tensile, True
Strength, Tensile, Ultimate
Stress, General
Stress, Tensile
Tenacity, Breaking
Toughness
Toughness, Breaking
Unevenness (U)
Wale
Water Resistance, *Fabric*
Water Repellency, *Textile*
Weight *n. Warp Knitting*
Yield, *n. Warp Knitting*

The committee recommends that the tentative revisions[4] as indicated of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

Specifications and Method of Test for Asbestos Roving for Electrical Purposes (D 375 – 58) (Sections 4, 5, and 9), and

Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics (D 462 – 53) (Section 9).

Revision of Standards, Immediate Adoption

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard General Methods of Testing Woven Fabrics (D 39 – 49):

Section 15.—Delete the Trapezoid Method.

Section 16.—Renumber as Section 15, and change to read as follows:

> 15. *Tongue Method:* (*a*) *Apparatus.*—For this single-rip tongue tear test procedure, use either a constant-rate-of-elongation or constant-rate-of-traverse tension testing machine as described in the Specifications for Textile Testing Machines (ASTM Designation: D 76).

---

[4] 1958 Book of ASTM Standards, Part 10, pp. 167 and 220.

(*b*) *Test Specimens.*—The test specimens shall be 3 in. in width and at least 8 in. in length. Two sets of five specimens each are required, one set for warp tearing strength, having the longer dimension parallel to the filling yarns, and the other set for filling tearing strength, having the longer dimension parallel to the warp. A longitudinal cut 3 in. in length shall be made lengthwise of the specimen starting in the center of one of the short edges.

(*c*) *Procedure.*—Place the specimen in the machine with one of the tongues in each clamp. Start the machine and observe the average load necessary to tear the fabric, preferably by means of an autographic recording device. Report the average of the results of the five individual tests on the warp as the warp tearing strength, and the average of the five individual tests on the filling as the filling tearing strength.

16. *Falling Pendulum Method.*—The tear resistance of woven fabrics may also be determined by the falling pendulum (Elmendorf) apparatus as described in the Method of Test for Tear Resistance of Woven Fabrics by Falling-Pendulum (Elmendorf) Apparatus (ASTM Designation: D 1424).

Footnote 4.—Add the reference to Footnote 4 against "C. *Tearing Strength*" with the footnote to read as follows:

> [4] The trapezoid method, formerly described in Methods D 39 - 49, has been rescinded since it is essentially the sort of tension test which is affected only by the properties of the yarns that are gripped in the jaws, and not by the fabric structure.

Standard Methods of Testing Felt (D 461 - 57): Revise as appended hereto.[5]

Standard Methods of Testing Nonwoven Fabrics (D 1117 - 58):

New Sections.—Add methods for crease resistance and drycleaning as new Sections 14 and 15, as appended hereto.[5]

Standard Method of Test for Fineness of Cotton Fibers by Micronaire (D 1448 - 58): Revise as appended hereto.[5]

---

[5] The revised standards appear in the 1959 Supplement to Book of ASTM Standards, Part 10.

REPORT OF COMMITTEE D-14
ON
ADHESIVES*

Committee D-14 on Adhesives held two meetings during the year: at the Shoreham Hotel, Washington, D. C., on October 16 and 17, 1958, and at the School of Natural Resources, University of Michigan, Ann Arbor, Mich. on April 28 and 29, 1959.

The committee consists of 131 members, of whom 46 are classified as producers, 63 as consumers, and 22 as general interest members.

West Coast sections were established for Subcommittee X on Wood Bonding and for Subcommittee XI on Metal Bonding.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of all of the tentatives under its jurisdiction.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Tests (D. K. Rider, chairman) has prepared a method for climbing drum peel test for adhesives. Work is being undertaken on effect of loading rate on T-peel strength prior to presenting the method for committee ballot.

Revisions of the rate of loading to be specified in the Method of Test for Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) (D 1002 – 53 T) are being submitted to the committee. Tensile, torsional shear, impact, and cleavage methods based on butt rod joints were reviewed for consideration by the subcommittee. A disk shear test was also described.

Subcommittee II on Analytical Tests (G. F. Lipsey, chairman) has prepared a method for the non-volatile content of solvent-type adhesives. A method for the specific gravity of solvent-type adhesives is being submitted to the committee.

Subcommittee III on Tests for Permanency (H. R. Butzlaff, chairman) has prepared for presentation to the committee a recommended practice for exposure of adhesive specimen materials to high-energy radiation. A report was prepared on the results of an interlaboratory test program which showed that adherends other than wood were suitable for testing according to the Tentative Methods of Test for Resistance of Adhesives for Wood to Cyclic Laboratory Aging Conditions (D 1183). A revision of the method has been prepared. The subcommittee is preparing a method on the mold resistance of plywood glues. Work has been started to develop an over-all plan for conducting exposure tests of adhesive-bonded specimens at atmospheric exposure test sites.

Subcommittee IV on Working Qualities (N. J. DeLollis, chairman) has prepared a method for the determination of pressure-sensitive tack and for the flow of adhesives. A method on determination of consistency by penetration is being ballotted by the subcommittee. Methods on the determination of consistency by the Zahn cup and by the Saybolt apparatus are being drawn up. The change of tack with time is being discussed to determine whether a standard test method can be written.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

Subcommittee V on Specifications (M. Petronio, chairman) is completing a specification for polyvinyl acetate resins emulsion adhesives and for general purpose (mending) adhesives.

The specification on adhesives for acoustical materials has been revised and is ready for presentation to the committee.

The specifications on sealing of top flaps, water absorptiveness, and bonding permanency of adhesives for fiberboard shipping cases and the revision of the adhesive sampling procedure are being revised based on comments from letter ballots of the subcommittee.

Subcommittee VI on Nomenclature and Definitions (F. H. Wetzel, chairman) prepared definitions of "feathering" and "throwing" for committee consideration, and definitions of "mastic," "cobwebbing," "body," and "cottoning" for subcommittee consideration.

Subcommittee VII on Research (C. B. Hemming, chairman) presented an informal session on adhesion testing at the April meeting in Ann Arbor. J. E. Marian, California Forest Products Laboratory, discussed "Surface Properties of Wood as Related to Joint Strength," and J. E. Rutzler, Jr., Case Institute, presented "Surface Properties of Metals as Related to Joint Strength."

Subcommittee X on Wood Adhesives (R. F. Blomquist, chairman) is now organized into two groups, based on geographical locations and interests, for greater efficiency. The East Coast group, under the chairmanship of R. F. Blomquist, is composed of individuals primarily in the East, South, and Middle West where interests include hardwood plywood, laminating, and furniture and specialty fabrication. The West Coast group, of which J. R. Ash is chairman, consists primarily of individuals in the Pacific Northwest and in California where softwood plywood and laminating are of greatest interest. Both groups will have similar organizations including sections on lumber adhesives, veneer adhesives, assembly adhesives, pulp and particle board binders, adhesives for wood to other materials, and special problems. A Bond Quality Symposium was held at Westminister, B. C., on October 28. 1958, by the West Coast group, at which time the following subjects were informally presented: "Some Processes in Testing End-Wood Glue Joints" by J. E. Marian; "Cycling-to-Delamination Method" by P. L. Northcott; "Industrial Uses of an Automatic Machine for Glue Bond Testing" by B. S. Bryant and O. F. Campbell, Jr.; and "A Modified Interior Test Procedure" by G. Sisterbenm. The East Coast group is circulating a questionnaire among present Committee D-14 members and other potential members to determine their interest in the activities of the new group and to determine the most important test procedures needed for wood adhesives.

Subcommittee XI on Metal Bonding Adhesives (F. H. Bair, chairman) is reviewing existing test methods, and new methods are being developed for shear, creep, fatigue, impact, high- and low-temperature testing, weathering exposure, permanence in exposure to various media, metal surface preparation, and inspection and process control.

A West Coast task group has been organized to consider some of the test methods. Three meetings have been held in which considerable progress has been made toward development of methods. Stephen Yurenka, Narmco Industries, Inc., San Diego, Calif. is chairman of the West Coast group.

Subcommittee XII on Adhesives for Plastics (D. A. George, chairman) has set up Section A on Adhesives for Reinforced Plastics. A questionnaire has been sent to the subcommittee membership inquiring as to their interest in test methods for adhesives used to bond phenolics, ureas, polyesters, epoxies, melamines, vinyls, methacrylates, cellulosics, polystyrene, and polyethylene. Information is also required as to permanence tests for these applications.

This report has been submitted to letter ballot of the committee, which consists of 114 members; 75 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. E. RUTZLER, JR.,
Chairman.

J. J. LAMB,
Secretary.

REPORT OF COMMITTEE D-15
ON
ENGINE ANTIFREEZES*

During the year Committee D-15 on Engine Antifreezes held one meeting: on April 14, 1959, in Washington, D. C. Subcommittee meetings were held on October 23, 1958, in Philadelphia, Pa., and on April 12, 13, and 14, 1959, in Washington, D. C.

Two members resigned and three members were elected to membership in Committee D-15 during the year. The committee now has 37 members, of whom 12 are classified as producers, 15 as consumers, and 10 as general interest members.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as appended hereto[1] of the Standard Method of Test for Water in Concentrated Antifreezes by the Iodine Reagent Method (**D 1123 – 54**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method for Glassware Corrosion Test for Engine Antifreezes (**D 1384 – 55 T**).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Physical Properties (C. M. White, chairman) by an interlaboratory collaborative study has found that Method D 1540 on Effect of Staining Agents on Organic Finishes can be used to evaluate dyes in engine antifreezes.

Subcommittee IV on Chemical Properties (E. F. Harford, chairman) has revised the Method of Test for Water in Concentrated Engine Antifreezes by the Iodine Reagent Method (D 1123 – 54) to replace the methanol base reagent with the glycol ether base reagent used in Method D 1364 – 55 T.

The Study Group on Glycol Analysis recommended gas chromatography as the most promising method for separation of monoglycols and polyglycols.

Subcommittee V on Effect of Antifreeze on Rubber Hose (O. W. Chandler, chairman) is considering three methods for evaluating the effect of antifreezes on cooling system hoses: a simulated service test, a bench-type test to measure change in properties of elastomers, and a test to determine change in burst strength caused by exposure to antifreezes.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The revised standard appears in the 1959 Supplement to Book of ASTM Standards, Part 8.

[2] 1958 Book of ASTM Standards, Part 8.

[3] The results of the letter ballot vote were favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VI on Simulated and Actual Service Testing (C. H. Sweatt, chairman).—The Study Group on Glassware Test Methods (E. L. Gibson, chairman) is instituting a third interlaboratory study on a laboratory screening method for foaming tendency. A collaborative test is also being initiated to evaluate proposed changes in the glassware corrosion test (Method D 1384 – 55 T).

The Study Group on Service Simulation Test Methods (V. O. Hatch, acting chairman) is continuing the development of apparatus and operating procedure for a bench-type circulating test that would evaluate corrosion and foaming properties under simulated service conditions. Two prototype assemblies have been in operation. Members are in the process of purchasing and setting up standardized apparatus. This apparatus consists of the basic components of an automotive cooling system including a cast iron reservoir, an automotive radiator, and coolant pump. A tentative operating procedure has been established.

Subcommittee VII on Specifications (B. E. Tiffany, chairman) is working on a specification and a recommended practice for purchasing engine antifreezes.

Subcommittee VIII on Editorial and Coordination of Test Methods (G. Kaufman, chairman) has reviewed and advised on the revision of Method D 1123 presented earlier in this report.

This report has been submitted to letter ballot of the committee which consists of 37 voting members; 24 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. VOGEL,
Chairman.

C. O. DURBIN,
Secretary.

REPORT OF COMMITTEE D-16
ON
INDUSTRIAL AROMATIC HYDROCARBONS AND RELATED MATERIALS*

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials and its subcommittees held two meetings during the year: on June 26 and 27, 1958, in Boston, Mass., and on February 4 to 6, 1959, in St. Louis, Mo.

Two new subcommittees were formed: Subcommittee H on American Group ISO/TC 78 on Aromatic Hydrocarbons with K. M. Brown as chairman, and Subcommittee I on Phthalic Anhydride with O. E. Knapp as chairman.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee D-16 presented to the Society through the Administrative Committee on Standards the recommendation that the Method of Test for Water in Phenol and Related Materials by the Iodine Reagent Method (D 1631 - 59 T) be published as tentative.[1] This recommendation was accepted by the Standards Committee on March 25, 1959, and the new tentative will appear in the 1959 Supplement to the Book of ASTM Standards, Part 8.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the proposed Method of Test for *o*-Xylene, *m*-Xylene, *p*-Xylene, and Ethylbenzene in Nitration Grade Xylene by Infrared Spectrophotometry.[2] This method was developed in the belief that industry needed a rapid means of determining the isomer distribution of nitration grade xylene.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods of test:[3]

Tentative Methods of Test for:

Color of Solid Aromatic Hydrocarbons and Related Materials in the Molten State (Platinum-Cobalt Scale), and

Traces of Thiophene in Benzene Using Isatin and Spectrophotometry.

The proposed Method of Test for Traces of Thiophene in Benzene Using Isatin and Spectrophotometry is a revision of the method published as an Appendix to the 1956 Compilation of ASTM Standards on Benzene, Toluene, Xylene, and Solvent Naphtha. A table of supporting data for this proposed method of test appears in Appendix III.[4]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Specific Gravity of Industrial Aromatic Hydrocarbons (D 891 – 51),[5] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

[1] Cooperative data for this method are given in Appendix I, p. 509.

[2] See p. 510.

[3] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 8.

[4] See p. 514.

Section 6(a).—Delete and replace with the following:

6. (*a*) *Hydrometer.*—The hydrometers to be used shall be those specified in Method E 100 as follows:

Nominal Specific Gravity Range	Hydrometer
0.850 to 0.900	86 H – 58 T
0.900 to 0.950	87 H – 58 T
0.950 to 1.000	88 H – 58 T

NOTE.—Corresponding hydrometers calibrated with the API scale may be used and the API reading converted to specific gravity with Table 3 of ASTM-IP Petroleum Measurement Tables (D 1250). In case of dispute the 86H, 87H, and 88H hydrometers shall be controlling.

This revision was requested by Subcommittee 18 on Hydrometers of Committee E-1 on Methods of Testing.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Bromine Index of Aromatic Hydrocarbons by Coulometric Titration (D 1492 – 57 T).[5]

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following tentatives:

Tentative Method of Test for Bromine Index of Aromatic Hydrocarbons by Potentiometric Titration (D 1491 – 57 T):[5]

New Section.—Add a new Section 2 entitled "Definition," to read as follows, renumbering the subsequent sections accordingly:

2. *Bromine Index.*—The number of milligrams of bromine consumed by 100 g of sample under given conditions.

Tentative Method of Test for Solidification Points of Chemicals (D 1493 – 57 T):[5]

Title.—Revise the title to read "Tentative Method of Test for Solidification Point of Aromatic Hydrocarbons and Related Materials."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A on Monocyclic Aromatics (S. S. Kurtz, Jr., chairman) prepared the proposed Method of Test for Traces of Thiophene in Benzene Using Isatin and Spectrophotometry; the table of supporting data for this method; the revision of Method D 891; the editorial revision of Method D 1491; and the Proposed Method of Test for *o*-Xylene, *m*-Xylene, *p*-Xylene, and Ethylbenzene in Nitration Grade Xylene by Infrared Spectrophotometry referred to earlier in the report.

This subcommittee is working on revisions of the Method of Test for Solidification Point of Benzene (D 852 – 47), the Methods of Test for Acid Wash Color of Benzene, Toluene, Xylenes, Refined Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons (D 848 – 47), and the Tentative

---

[5] 1958 Book of ASTM Standards, Part 8.

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene (D 1555 - 58 T). Also under consideration are a revision of the Method of Test for Paraffins in Industrial Aromatic Hydrocarbons (D 851 - 47), a method for nonvolatile contamination of industrial benzene, toluene, and xylene, a method for parts per million carbon disulfide in benzene, methods and specifications for styrene, a revision of the Method of Test for Distillation of Industrial Aromatic Hydrocarbons (D 850 - 56), and methods for traces of chlorides and sulfur in aromatic hydrocarbons.

Subcommittee B on Polycyclic Aromatics (M. Mitchell, chairman) collaborated with Subcommittee C in preparing the Method of Test for Color of Solid Aromatic Hydrocarbons and Related Materials in the Molten State (Platinum-Cobalt Scale) referred to earlier in the report, and is negotiating with the National Bureau of Standards for the procurement of thermometric reference cells for naphthalene, phthalic anhydride, and phenol. Subcommittee B is also considering methods for sulfur in naphthalene and the acid wash color (or substitute therefor) of naphthalene, and a specification for crude naphthalene.

Subcommittee C on Phenolic Compounds (D. F. Pontz, chairman) prepared the Method of Test for Water in Phenol and Related Materials by the Iodine Reagent Method and collaborated with Subcommittee B in preparing the Method of Test for Color of Solid Aromatic Hydrocarbons and Related Materials in the Molten State (Platinum-Cobalt Scale). The subcommittee is also working on methods for tar acids, phenol assay, and the water solubility of refined phenol.

Subcommittee D on Nitrogen Heterocyclics (R. F. Shertzer, chairman) is working on methods and specifications for refined pyridine and quinoline.

Subcommittee E on Thermometry (M. Lapeyrouse, chairman) is working with Subcommittee B on thermometric reference standards and is also studying: (1) problems connected with the use of wide range partial immersion thermometers on narrow boiling range fractions, (2) the calibration of partial immersion thermometers under conditions of use by observing the 50 per cent point of pure compounds (and the barometric pressure), and (3) the possible use of the platinum resistance thermometer for calibration of ASTM thermometers under conditions of use.

Subcommittee F on Editorial and Nomenclature (E. T. Scafe, chairman) is considering safety precautions for committee methods and is functioning as a consultant body to aid in the programs of the other subcommittees.

Subcommittee G on Statistical Procedure (W. J. Tancig, chairman) is revising its interlaboratory testing program, evaluating other more complex approaches to the calculation of repeatability and reproducibility, and is serving as a consultant body to aid the other subcommittees when statistical problems are involved.

Subcommittee H on American Group ISO/TC 78 on Aromatic Hydrocarbons (K. M. Brown, chairman) is awaiting formal action by ASA on the Committee D-16 recommendation that ASA elect participating status in ISO/TC 78.

Subcommittee I on Phthalic Anhydride (O. E. Knapp, chairman) has already begun active study of methods and specifications for this product.

This report has been submitted to letter ballot of the committee, which consists of 62 voting members; 46 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. E. SISCO,
Chairman.

K. H. FERBER,
Secretary.

APPENDIX I

COOPERATIVE DATA

Table I represents data obtained by cooperators using the modified Karl Fischer method of analysis for water in phenol.

TABLE I.—COOPERATIVE DATA ON METHOD FOR WATER IN PHENOL.

Sample No.[a]	Water, per cent by weight							
	1	2	3	4	5	6	7	8
Laboratory								
A	0.028 0.027	0.094 0.095	0.010 0.010	0.036 0.036	0.066 0.066	0.112 0.115	0.019 0.019	0.047 0.047
B	0.034 0.034	0.099 0.101	0.009 0.010	0.030 0.028	0.061 0.063	0.114 0.113	0.018 0.018	0.046 0.045
C	0.046 0.047	0.109 0.111	0.013 0.014	0.029 0.028	0.067 0.067	0.118 0.116	0.017 0.017	0.043 0.042
D	0.072 0.073	0.108 0.108	0.025 0.025	0.041 0.045	0.075 0.078	0.129 0.129	0.045 0.046	... ...
E	0.047 0.047	0.102 0.104	... ...	0.040 0.042	0.069 0.069	0.116 0.118	0.032 0.035	0.052 0.053
F	0.049 0.051	0.120 0.117	0.016 0.016	0.042 0.044	0.073 0.078	0.122 0.121	0.027 0.029	0.049 0.049
Average	0.046	0.106	0.015	0.037	0.069	0.119	0.027	0.047

[a] Odd numbered samples were production-grade phenol. Even numbered samples were contaminated with water specified not to exceed 0.1 per cent.

APPENDIX II

PROPOSED METHOD OF TEST FOR *ORTHO*-XYLENE, *META*-XYLENE, *PARA*-XYLENE, AND ETHYLBENZENE IN NITRATION GRADE XYLENES BY INFRARED SPECTROPHOTOMETRY[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope and Application

1. This method is intended for the determination of *ortho-*, *meta-*, and *para*-xylene and ethylbenzene in nitration grade xylenes. It is not recommended for the determination of the ethylbenzene content of samples containing less than 5 per cent ethylbenzene.

Summary of Method

2. The baseline absorbance of a blend of pure xylene isomers having a composition typifying nitration grade xylene is determined at four wavelengths in the 12 to 15-μ region, using a recording infrared spectrophotometer. A relatively nonabsorbing solvent is used to dilute the sample to a concentration that permits accurate absorbance measurements. The baseline absorptivity of each component of the blend of pure isomers is calculated. The unknown is then measured in the same manner and the concentration of each isomer in the sample is calculated.

Definitions

3. (*a*) *Transmittance*, *T*, is the ratio of the radiant power transmitted by the sample, *P*, to the radiant power incident on the sample, P_o, both being measured at the same spectral position and with the same slit width.

$$T = \frac{P}{P_o}$$

Under the conditions of this method:

$$T = 10^{-abc}$$

where *a*, *b*, and *c* are as defined in Paragraphs (*c*) to (*e*).

(*b*) *Absorbance*, *A*, is the logarithm to the base 10 of the reciprocal of the transmittance.

$$A = \log_{10} \frac{1}{T} = abc$$

(*c*) *Absorptivity*, *a*, is the ratio of the absorbance to the product of the concentration and the length of optical path.

$$a = \frac{A}{bc}$$

(*d*) *Length of Optical Path*, *b*, is the distance in millimeters traversed by the beam of radiation in passing through the sample.

(*e*) *Concentration*, *c*, is the quantity of absorbing substance per unit volume. In this method, *c* is expressed in grams per milliliter.

---

[1] This proposed method is under the jurisdiction of the ASTM Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials.

[2] Published as information, November, 1959.

(*f*) *Baseline Absorbance, A′*, is calculated as follows: A P_o line is drawn between designated points, which will usually be absorption minima or band shoulders. A perpendicular to the abscissa (wavelength axis) of the spectrum is erected so as to pass through the absorption peak (or other analytical wavelength) and intersect the P_o line (baseline). The absorbance at the intersect is subtracted from the absorbance at the peak (or other analytical wavelength) to obtain the baseline absorbance.

$$A' = A_{peak} - A_{baseline}$$

Apparatus

4. (*a*) *Infrared Spectrophotometer.*—Any recording infrared spectrophotometer having the necessary stability, reproducibility, and resolution in the 12 to 15-μ region. The spectrophotometer shall be capable of repeating absorbances at the 0.5 level with a repeatability of ±0.5 per cent from an average in this spectral region and shall be designed so that the wavelength settings are reproducible to 0.01 μ or better.

(*b*) *Sample Cell*, made of sodium chloride or potassium bromide, and having an optical path length of about 0.1 mm.

NOTE 1.—The same absorption cell must be used for both calibration and determination.

(*c*) *Volumetric Flasks*, 25-ml, calibrated, glass-stoppered.

Solvent

5. *Cyclohexane or Carbon Disulfide*, free of aromatics, may be used as a spectroscopic solvent. The same solvent must be used in the analysis as was used in calibration.

Reference Standards

6. Samples of each of the xylene isomers being determined, having a minimum purity of 99 per cent, shall be used as reference standards. Only hydrocarbons in which the composition of the impurities is known are suitable unless the impurity is less than 0.1 per cent.

NOTE 2.—American Petroleum Institute standard samples may be used.

Calibration of Spectrophotometer

7. (*a*) *Selection of Slit Width.*—Choose a slit program such that the signal-to-noise ratio is at least 200 at each of the selected spectral positions (Paragraph (*b*)) so that duplicate measurements of absorbance are reproducible to ±0.5 per cent from the average.

(*b*) *Measurement of Absorbance of Standard.*—Determine the exact spectral position for each of the compounds listed in Table I by finding the wavelength of maximum absorption nearest the listed spectral position, using a blend of the pure isomers that will approximate in percentage by weight composition a typical nitration grade xylene. Follow the procedure described in Section 8.

TABLE I.—SPECTRAL POSITIONS.

Compound	Spectral Position, μ
o-xylene	13.47
m-xylene	13.01
p-xylene	12.58
ethylbenzene	14.36

NOTE 3.—For instruments having potassium bromide optics, a position of 17.97 μ is a desirable alternative for ethylbenzene.

NOTE 4.—If a separate peak is not resolved for ethylbenzene, the ethylbenzene wavelength may be adequately defined in terms of a distance from the *m*-xylene peak near 14.5 μ as follows:

Analytical wavelength = observed location of the *m*-xylene peak near 14.5 μ − 0.12 μ.

Using the slit program determined, record the infrared spectrum from 12 to 15 μ. At each of the determined spectral positions, measure the baseline absorbance of each component after constructing the following baselines:

For *o*-, *m*-, and *p*-xylene, draw a straight line connecting the absorption minimum near 12.3 μ with that near 14.0 μ.

For ethylbenzene (14.36 μ peak), draw a straight line connecting the absorption minimum near 14.0 μ with that near 15.0 μ.

(*c*) *Calculation of Absorptivities.*—Calculate the absorptivity, *a*, of each component as follows:

$$a = \frac{A'}{bc}$$

where:

b = length of optical path in millimeters, but can be assumed equal to unity since the same absorption cell is used for both calibration and determination, and

c = concentration in grams of blend per milliliter of solution (for each isomer) multiplied by the fraction by weight of that isomer in the blend.

NOTE 5.—Unless the spectrophotometer is equipped with a thermostatted cell compartment, precautions must be taken to insure that the temperature at which determinations are made does not differ from the calibration temperature by more than 3 C.

Procedure

8. Take a 25-ml volumetric flask partly filled with spectroscopic solvent. Introduce about 1 ml of sample and accurately determine the weight of the sample. Fill the flask to the calibration mark with spectroscopic solvent and determine the baseline absorbance at each position as described in Section 7.

Calculations

9. (*a*) Calculate the percentage by weight of each isomer in the sample as the quotient of the sample baseline absorbance by the product of the corresponding standard absorptivity (as determined in accordance with Section 7 (*c*)) and the grams of sample per milliliter of solution. For example:

o - xylene, per cent by weight

$$= \frac{A' \text{ at } 13.47\ \mu \text{ for sample}}{a \text{ at } 13.47\ \mu \text{ for } o\text{-xylene} \times c}$$

where:

c = grams of sample per milliliter of solution.

(*b*) Normalize the results, expressed as per cent, to total 100 minus the percentage of paraffins, where the percentage of paraffins is determined in accordance with the Method of Test for Paraffins in Industrial Aromatic Hydrocarbons (ASTM Designation: D 851).[3] For example:

o-xylene, normalized, per cent

$$= \frac{(100 - X) \times Y}{Z}$$

where:

X = percentage of paraffins,

Y = percentage of *o*-xylene found, and

Z = total percentage of isomers found.

(*c*) In the event that one or more of the normalized percentages differs from the corresponding percentage for that isomer in the standard blend by more than 3 per cent absolute, the determination must be repeated using for calibration a new standard blend of pure isomers for calibration such that the composition of the new standard agrees within 1 per cent absolute for each isomer of the apparent composition of the sample.

Repeatability and Reproducibility

10. (*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts (expressed in normalized per cent):

o-xylene	1.1
m-xylene	3.7
p-xylene	2.4
ethylbenzene	2.1

(*b*) *Reproducibility.*—The result submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts (expressed in normalized per cent):

---

[3] 1958 Book of ASTM Standards, Part 8.

o-xylene	1.1
m-xylene	3.7
p-xylene	2.4
ethylbenzene	4.6

NOTE 6.—The values for repeatability and reproducibility given in Section 10 are based upon a collaborative test by seven laboratories upon a sample having the following approximate composition:

o-xylene	10 per cent
m-xylene	57 per cent
p-xylene	25 per cent
ethylbenzene	7 per cent

The estimates of repeatability and reproducibility given in Section 10 apply only to such a composition. However, the restriction concerning the composition of the standard blend was not then imposed. It is believed that the application of the present draft of the method, which reduces that source of interlaboratory variability, will result in a lower value for reproducibility, particularly in the case of ethylbenzene.

APPENDIX III

RESULTS ON THIOPHENE IN BENZENE COOPERATIVE PROGRAM USING PROPOSED METHOD

	ppm Thiophene				
Sample No. Calculated Content	J-1069 0.00	J-1070 0.46	J-1071 4.8	J-1072 57	J-1073 228
Laboratory:					
A	0.01	0.43	5.4	55	236
	0.02	0.39	5.4	53	230
	0.01	0.37	4.7	50	253
	—	—	—	—	—
	0.01	0.40	5.2	53	240
B	<0.1[a]	0.5[a]	5.2	62	223
	<0.1	0.5	5.2	60	219
	<0.1	0.5	5.2	61	221
C	nil[a]	0.47	4.9	57	232
D	0.00	0.47	4.6	54	238
	0.01	0.49	4.6	53	242
	0.01	0.46	4.3	55	218
	—	—	—	—	—
	0.01	0.47	4.5	54	233
E	0.06	0.53	4.6	60	221
	0.04	0.51	4.7	55	238
	—	—	—	—	—
	0.05	0.52	4.7	58	230
F	<0.02	0.47	4.8	50	214
	<0.02	0.48	4.8	53	214
	0.04	0.43	5.1	54	214
	—	—	—	48	—
	~0.03	0.46	4.9	—	214
				51	
G	0.03	0.52	4.1	55	223
	0.03	0.49	4.5	55	223
	0.03	0.52	4.5	52	223
	—	—	—	—	—
	0.03	0.51	4.4	54	223
H	0.01	0.46	4.7	57	215
	0.01	0.46	4.7	58	215
	0.01	0.48	4.7	55	215
	—	—	—	—	—
	0.01	0.47	4.7	57	215

		Per cent of Value		Per cent of Value		Per cent of Value		Per cent of Value		Per cent of Value
Average (of laboratory averages)	0.02		0.47		4.8		56		226	
Difference of average from calculated value	0.02		0.01		+0.0		+1		+2	
Repeatability:										
Sum of squares	0.0007		0.0049		0.57		64		769	
Degrees of freedom	11		11		12		13		12	
Standard deviation	0.008	40	0.021	4.5	0.22	4.6	2.2	3.9	8.0	3.5
95 per cent confidence range	0.025	125	0.065	13.8	0.68	14.2	6.7	12.0	25	11.1
Reproducibility:										
Sum of squares	0.0014		0.0091		0.61		73		596	
Degrees of freedom	5		6		7		7		7	
Standard deviation	0.017	85	0.039	8.3	0.30	6.3	3.2	5.7	9.3	4.1
95 per cent confidence range	0.062	310	0.135	28.7	1.01	21.1	10.7	19.1	31	13.7

---

[a] Results omitted in calculation of repeatability and reproducibility.

REPORT OF COMMITTEE D-17
ON
NAVAL STORES*

Committee D-17 on Naval Stores held one meeting during the year: on June 26, 1958, at Boston, Mass., in connection with the Annual Meeting of the Society.

The committee regrets to report the loss through death of J. Mack Schantz, technical assistant to the manager of pine chemicals in naval stores, Hercules Powder Co., one of its long-time collaborating company representatives. He will be replaced by J. N. Borglin. Other members resigning from the committee were K. A. Perkins, of National Southern Products Co., and R. P. Chapman and R. P. Smith of American Cyanamid Co.

The special committee working on revisions of Federal Specifications for Naval Stores, on which the Society is represented by the Chairman of Committee D-17, has completed the revision in the interim form of the Specification for Pine Oil (LLL-P-00400) and the Specification for Pine Tar (LLL-P-00430).

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Methods of Test for Saponification Number of Rosin (D 464 - 51):[1]

Section 5.—Add a new Paragraph (*e*) to read: "(*e*) *Thymol Blue Indicator Solution.*—Dissolve 0.1 g of thymol blue in 100 ml of alcohol conforming to Paragraph (*a*)."

Section 6 (*d*).—Change to read as follows:

> (*d*) Cool the solutions and titrate with the standard sulfuric acid using 1 ml of either phenolphthalein or thymol blue indicator. With thymol blue, the end point is indicated when the color undergoes the first change from a distinct blue to a yellowish-green, just short of clear yellow.

Section 8 (*a*).—Add the following sentence: "Alternatively an automatic potentiometric titrator may be used."

Section 11.—Add a new Paragraph (*f*) and a Note to read as follows:

> (*f*) Alternatively, if an automatic titrator is used, the end point shall be taken either as the inflection point from the plotted curve or a definite pH value (Note), depending on the type of instrument.
>
> NOTE.—This pH value should be determined by plotting the titration curve for several typical samples of the same type rosin under closely controlled conditions, type of solvent, water ratio, etc. Use the average of the pH values at the inflection point. Glass electrodes tend to dehydrate in nearly anhydrous solvent medium. Condition the electrode in water between tests and check with known pH buffers frequently.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] 1958 Book of ASTM Standards, Part 8.

Standard Methods of Test for Acid Number of Rosin (D 465 - 51):[1]

Section 5 (*b*).—Change to read as follows:

(*b*) Heat if necessary to dissolve the rosin, cool to room temperature (Note), and titrate the solution with the standard alkali solution using 1 ml of either phenolphthalein or thymol blue indicator. With phenolphthalein, titrate to the first persistent faint pink color; with thymol blue, titrate to a distinct blue.

Section 7 (*a*).—Add the following sentence: "Alternatively, an automatic potentiometric titrator may be used."

Section 10 (*d*).—Add the following sentence and Note to read as follows:

Alternatively, if an automatic titrator is used, the end point shall be taken either as the inflection point from the plotted curve or pH 10.8 (Note) with instruments which titrate to a preset value.

NOTE.—The value of 10.8 is the average pH encountered at the inflection point by the above procedure using closely controlled conditions, solvent, water ratio, etc. Glass electrodes tend to dehydrate in nearly anhydrous solvent medium. Condition the electrode in water between tests and check with known pH buffers frequently.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Softening Point of Rosin (J. N. Borglin, chairman) has continued to evaluate methods for determining the melting points of various rosins. Presently comparative tests are under way wherein the Hercules drop method is being compared with the Newport capillary tube method and also the ASTM ring and ball method. To insure maximum utility of results, four types of rosin are being used: namely, FF wood rosin, tall oil rosin, a higher melting modified wood rosin, and finally gum rosin.

Subcommittee II on Crystallization of Rosin (R. V. Lawrence, chairman) has made further comparisons of the method developed by S. R. Snider[3] with that of Palkin and Smith[4] and the Bakelite[5] method which confirmed the work reported last year. The method developed by Snider appeared to be more sensitive than the other two. The Palkin and Smith method is simpler and gives results more quickly. It was found that the Palkin-Smith method did not require seeding. Twelve samples of gum rosin requiring from one hour to more than two days to form crystals were examined and in no case did the addition of seed decrease the length of time required to form crystals or to produce crystals where none were obtained without seed. Apparently the range of concentrations obtained with this method is great enough to provide the optimum conditions for crystal formation.

In order to correlate actual plant difficulties from rosin crystallization with present methods of testing, attempts were made to obtain samples of gum rosin that gave trouble from crystallization in industrial processing but no such samples have been obtained.

It has been recommended that when this study is completed, a summary of the work be published in a U. S. Department of Agriculture bulletin.

Subcommittee IV on Chemical Analyses of Rosin (R. Herrlinger, chairman) has recommended for adoption the revisions in Methods D 464 and D 465 referred to earlier in the report.

A new collaborative study was made comparing the Wolff potentiometric method, the Wolff colorimetric method, and the Linder-Persson method for the determination of fatty acids in tall oil rosin. The agreement between operators and laboratories was poor. Additional collaborative work and some fundamental studies of the problems involved are planned by members of the committee.

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 531 (1955).

[4] *Journal*, Am. Oil Chemists' Soc., Vol. 15, p. 120 (1938).

[5] Private communication.

Subcommittee V on Tall Oil (A. Pollak, chairman) has been actively co-operating with various subcommittees and groups of Committee D-1 on Paint, Varnish, Lacquer, and Related Products. Collaborative work continues on the Tentative Method of Test of Total Rosin Acids Content of Coating Vehicles (D 1469 – 57 T). Members are not agreed on the advisability of conforming to the new Gardner-Holdt viscosity standards established in Committee D-1. Members also report resistance to the new Gardner color standards, and there seems to be some doubt on their reproducibility. There has been considerable discussion on the merit of reorganizing Methods D 803, especially since tall oil fatty acids testing is under the jurisdiction of Committee D-1.

Subcommittee VII on Terpene Hydrocarbons and Pine Oil (R. E. Price, chairman) is making a study of the feasibility of incorporating, as an alternate method, in the present distillation methods of turpentine, dipentene and pine oil, the use of the 200-ml distillation flask as described in Method D 1078. Previous collaborative tests have indicated the same results are obtained by either the 100- or 200-ml flasks.

At the present time there is only a general statement in regards to the color determination of pine oil in Methods D 802. It is proposed that a method be incorporated for color determination in Methods D 802 similar to those described in Methods D 801 and D 233.

Subcommittee IX on Definitions (J. L. Boyer, chairman) has recommended that the definitions for refined tall oil and tall oil fatty acids be the same as those adopted by the Tall Oil Division of the Pulp Chemicals Assn. Definitions for sulfate wood turpentine and rectified turpentine will also be submitted for the approval of Committee D-17.

Subcommittee X on Specifications (V. E. Grotlisch, chairman) has been considering the proposal of specifications for other naval stores (similar to the specification for turpentine), for approval and eventual adoption by Committee D-17, that would conform with those currently used by the Federal Government.

This report has been submitted to letter ballot of the committee, which consists of 31 voting members; 21 members returned their ballots, of whom 20 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. R. SNIDER,
Chairman.

W. A. KIRKLIN,
Secretary.

REPORT OF COMMITTEE D-18
ON
SOILS FOR ENGINEERING PURPOSES*

Committee D-18 on Soils for Engineering Purposes held its Annual Meeting in Boston, Mass., on June 27, 1958. Executive Subcommittee meetings were held on June 25, 1958, at Boston and on January 4, 1959, at Washington, D. C. A general Soils Session, consisting of six papers, was held during the Annual Meeting of the Society in Boston. These papers are published in the 1958 *Proceedings* of the Society. A Symposium on Application of Soil Testing in Highway Design and Construction was held in two sessions, at which time ten papers were presented.[1] A single paper on "Growth of Soil Testing in Southern Europe and the Middle East" was presented after the closing of the main meeting.

Committees have been appointed to secure papers for symposia on Rates of Loading in Soil Tests, and Atterberg Limits, to be presented at the 1959 Annual Meeting.[2]

A special committee, with F. J. Converse as chairman, has been appointed to secure papers for two or three soils sessions and to make other Committee D-18 arrangements for the Pacific Area National Meeting, San Francisco, October 1959.

Appointments as committee representatives on other committees are as follows: E. A. Abdun-Nur, on Committee E-11 on Quality Control of Materials; and C. R. Foster, on Committee E-8 on Nomenclature and Definitions.

The following members of Committee D-18 passed away during the year and will be missed greatly by their colleagues: F. R. Olmstead, A. E. Stoddard, and C. W. Allen.

Committee D-18 now consists of a total of 141 individual members and 18 consultants. There are 107 voting members, of whom 21 are classified as producers, 36 as consumers, and 50 as general interest members.

Special Committee for Hogentogler Award.—The committee has recommended that the Hogentogler Award be given to W. E. Schmid for his paper entitled "The Permeability of Soils and the Concept of a Stationary Boundary-layer," presented at the 1958 Annual Meeting.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee D-18 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of Test for:

Making and Curing Soil-Cement Compression and Flexure Test Specimens in the Laboratory (**D 1632 - 59 T**) (Joint with Committee D-4),

Compressive Strength of Molded Soil-Cement

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Issued as separate publication *ASTM STP No. 239.*

[2] Issued as separate publication *ASTM STP No. 254.*

Cylinders (**D 1633 – 59 T**) (Joint with Committee D-4),

Compressive Strength of Soil-Cement Using Portions of Beams Broken in Flexure (Modified Cube Method) (**D 1634 – 59 T**) (Joint with Committee D-4), and

Flexural Strength of Soil-Cement Using Simple Beam with Third-Point Loading (**D 1635 – 59 T**) (Joint with Committee D-4).

Revision of Tentative Definitions of:

Terms and Symbols Relating to Soil Mechanics (**D 653 – 42 T**).

These recommendations were accepted by the Standards Committee on March 25, 1959 with the exception of the revision of Definitions D 653, which was accepted on September 9, 1958. The new tentatives will appear in the 1959 Supplement to Book of ASTM Standards, Part 4. The revised tentative appears in the 1958 Book of ASTM Standards, Part 4.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Plastic Limit and Plasticity Index of Soils (**D 424 – 54 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without revision.

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Method of Test for Field Moisture Equivalent of Soils (**D 426 – 39**).[3]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following standards:

Tentative Methods of Test for:

Soil-Bituminous Mixtures (**D 915 – 47 T**) (Joint D-4),

Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus (**D 916 – 47 T**) (Joint D-4),

Load-Settlement Relationship for Individual Piles Under Vertical Axial Load (**D 1143 – 57 T**),

Water-Soluble Chlorides Present as Admixes in Graded Aggregate Road Mixes (**D 1411 – 56 T**) (Joint D-4), and

Soil Investigation and Sampling by Auger Borings (**D 1452 – 56 T**).

Tentative Specifications for:

Materials for Soil-Aggregate Subbase, Base, and Surface Courses (**D 1241 – 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

SUBCOMMITTEE REPORTS

Research Steering Committee (W. S. Housel, chairman).—A new subcommittee has been established for the administration and supervision of Committee D-18 research activities. This committee will hold its first meeting in June 1959.

Subcommittee G-2 on Special Papers (G. F. Sowers, chairman) has been appointed to secure papers for a Symposium on Soil Mechanics in Highway Design and Construction. This is a continuation of the symposium held last year on the same subject.

Subcommittee G-3 on Nomenclature and Definitions (C. R. Foster, chairman) concentrated on final publication of the Tentative Definitions of Terms and Symbols Relating to Soil Mechanics (D 653 – 58 T). This work has been done in coordination with the ASCE Committee on Glossary of Terms and Definitions on Soil Mechanics which published the coordinated glossary in the October 1958 Journal of the Soil Mechanics and Foundations Division to provoke further review. In the coming year, the subcommittee will continue to work with the ASCE committee toward a final published glossary and will take steps to have the ASTM Tentative adopted as standard.

---

[3] 1958 Book of ASTM Standards, Part 4.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee R-2 on Sampling and Related Field Testing for Soil Investigations (John P. Gnaedinger, chairman).—Revisions of the Method for Penetration Test and Split-Barrel Sampling of Soils and the Method for Thin-Walled Tube Sampling of Soils (D 1586 and D 1587) are under consideration.

The subcommittee is recommending that Method D 1452 be continued as a tentative pending possible revisions of the method.

A method of diamond core drilling has been prepared and will be discussed at the 1959 Annual Meeting. The subcommittee hopes to have a first draft of a method of vane shear testing this June.

The subcommittee is collaborating to the maximum extent possible with the Canadian Standards Assn. in an effort to establish an agreement on sampling methods between the two organizations.

Subcommittee R-3 on Physical Characteristics of Soils (E. E. Bauer, chairman) recommended (1) the discontinuance of Standard Method of Test for the Field Moisture Equivalent of Soils (D 426); (2) revisions of Tentative Method of Test for Liquid Limit (D 423) and Tentative Method of Test for the Grain-Size Analysis (D 422); and (3) adoption as standard of Tentative Method of Test for Plastic Limit and Plasticity Index (D 424). The revision of D 422 has been postponed until negative votes have been resolved.

Work is continuing on a proposed tentative method of wet preparation of soil samples for grain-size analysis and determination of soil constants by Sections A and B. Collection of available data for publication later concerning various phases of the moisture-density relations methods of test is being carried out by Section C. Phases include: sizes of molds, shapes of rammers, heights of drop, types of rammers, re-use of samples, and maximum sizes of particles used in test samples. The second phase of the testing program, in connection with the development of methods of test for the determination of maximum and minimum densities of granular soils, is being carried out by Section D.

A symposium on the Atterberg Limits is being sponsored by Section B at the 1959 Annual Meeting of the Society.[2]

Subcommittee R-4 on Physical Properties of Soils (N. W. McLeod, chairman) is working on drafts of tentative methods for measuring the capillarity potential of soils, and the coefficient of permeability of granular soils.

Subcommittee R-5 on Structural Properties of Soils (D. M. Burmister, chairman).—Some progress is being made in the preparation of standard methods of test for structural properties of soils. It is the consensus that specifications of apparatus should not be limited to a single type but should permit the use of most of the commonly used types and sizes of apparatus. The preparation of soil specimens is considered important, but no general agreement has been found. The loading conditions were recognized to be of principal importance and have been discussed at subcommittee meetings, but no consensus could be reached.

This subcommittee, together with Subcommittees R-2, R-8, R-10, and R-11, is presenting a Symposium on Time-Rates of Loading in Testing Soils[2] at the Annual Meeting, June, 1959. These papers may help to resolve some of the questions regarding the all-important subject of the influences of loading conditions in the strength testing of soils.

Subcommittee R-6 on Physico-Chemical Properties of Soils (D. T. Davidson, chairman).—The principal activity of this subcommittee during 1958 has been the planning of a monograph or manual on methods of evaluating physicochemical phenomena in soils. The fact that the Soil Science Society of America is planning a monograph on "Methods of Soil Analysis" has led to investigation of the desirability of ASTM cooperation. The tentative chapter outlines indicate that the subject coverage is more than adequate from an engineering standpoint. Opinions and recommendations from subcommittee members have been solicited.

Subcommittee R-8 on Special and Construction Control Tests (H. F. Clemmer, chairman).—The subcommittee held one meeting during the year with all of the sections reporting activity as follows:

Section A on Base Courses and Subgrades for Highways and Airports is continuing its consideration of the California Bearing Ratio method of determining the bearing of soils. It is expected that this method, as well as other bearing tests of soils, will be presented shortly.

Section B on Earth Dams and Embankments has given further consideration to the balloon method for determining density of soils and hopes to have a draft ready for consideration of the subcommittee.

Section C-1 on Cement presented the new tentative methods of test accepted by the Administrative Committee on Standards referred to earlier in the report.

Section C-2 on Bituminous has under consideration revisions to Methods D 915.

Section C-3 on Calcium Chloride is considering several changes in Specifications D 1241 to conform to changes made to a similar specification approved by AASHO. The section has also carried on considerable research on Methods D 916 and D 1411 with the possibility of recommending revisions in the future.

Section C-4 on Soil Conditioners has been largely concerned with formulating a program to be followed by the section. It is expected that such a program will be presented in the near future.

Subcommittee R-9 on Dynamic Properties of Soils (R. K. Bernhard, chairman) is working on a new tentative definitions of terms referring to soil dynamics.

Subcommittee R-11 on Pile Load Bearing Tests (W. S. Housel, chairman) has had under consideration a number of minor revisions in Methods D 1143, and recommends that the method be continued as tentative.

The subcommittee also reviewed the Suggested Method of Test for Load Capacity of Batter Pile Frames which has been included in the new edition of "Soil Test Procedures." This method will be discussed further in an effort to promote its publication as a tentative.

Joint Subcommittee D-4, D-18 on Tests and Specifications for Stabilized Soils (H. F. Clemmer, chairman) recommends that Methods D 915, D 916, and D 1411 and Specification D 1241 be continued as tentative pending revision.

This report has been submitted to letter ballot of the committee which consists of 107 voting members; 81 members returned their ballots, of whom 73 voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

E. J. KILCAWLEY,
Chairman.

W. G. HOLTZ,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-18 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Method for:

Grain-Size Analysis of Soils (D 422 – 54 T).

Revision of Tentative Method of Test for:

Liquid Limit of Soils (D 423 – 54 T).

These recommendations were accepted by the Standards Committee on November 9, 1959, and the revisions of the tentative methods appear in the 1959 Supplement to the Book of ASTM Standards, Part 4.

REPORT OF COMMITTEE D-19

ON

INDUSTRIAL WATER*

Committee D-19 on Industrial Water and its subdivisions held meetings June 25 to 27, 1958, in Boston, Mass., and on January 26 to 28, 1959, in Savannah, Ga.

The Symposium on Radioactivity in Industrial Water and in Industrial Waste Water sponsored by the committee and presented on June 26, 1958, in Boston, Mass., has been published as *ASTM STP No. 235*.

A Symposium on Identification of Water-Formed Deposits,[1] sponsored by the committee, will be presented on June 25, 1959, in Atlantic City, N. J. The following papers are to be included:

"Deposit Identification—The First Step Toward Understanding a Water Problem," by J. K. Rice, Cyrus William Rice & Co., Inc.

"Application of Emission Spectroscopy to the Analysis of Water-Formed Deposits," by Charles H. Anderson, Babcock & Wilcox Co.

"Identification by Instrumental Methods of Chemical Compounds in Water-Formed Deposits," by C. M. Maddin and R. B. Rosene, Dowell Division, The Dow Chemical Co.

"Electron Microscopy and Electron Diffraction Studies of Oxide Films Formed on Iron in Water Vapor and Oxygen Atmospheres," by Earl A. Gulbransen and Thomas P. Copan, Westinghouse Research Laboratories.

"Correlation of Elemental Analysis and Phase Identification as Viewed by a Mineralogist," by J. V. Smith, Pennsylvania State University.

"Summary," by R. K. Scott, Hays Laboratories, Harbison-Walker Refractories Co.

A Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water,[2] sponsored by the committee, will be presented at the Third National Pacific Area Meeting in October, 1959, in San Francisco, Calif. The following papers will be presented:

"Introduction," by W. L. Lamar, Quality of Water Branch, U. S. Geological Survey.

"Radioactive Waste Disposal at Hanford," by D. W. Pearce, C. E. Linderoth, R. E. Brown, L. C. Schwendiman, and J. F. Honstead, Hanford Atomic Products Operation.

"Determination of Radioactive Materials in Water," by F. B. Barker, Quality of Water Branch, U. S. Geological Survey.

"Improvements in Water Treatment for Once-Through Reactor Cooling," by R. B. Richman, Hanford Atomic Products Operation.

"Disposal of Oil Field Brines in the Central Valley of California," by James M. Morris, Jr., California Dept. of Water Resources.

"Cities Can and Must Provide Municipal Waste Treatment, Not Sewage Treatment," by J. E. Kinney, Sanitary Engineering Consultant.

"Field Testing of Corrosion Inhibitors in Sea Water," by C. C. Wright, Oilwell Research, Inc.

"Effect of Industrial Wastes on Waters for Irrigation Use," by L. V. Wilcox, U. S. Salinity Laboratory, U. S. Dept. of Agriculture.

"Operation of Batch-Type and Continuous Electric Membrane Demineralizers on the Municipal Water Supply of Coalinga, California," by W. E. Katz, Ionics Inc.

"Present Status of Solar Distillation of Saline Water, with Particular Regard to Material Problems (and Control of Evaporation Losses), by R. P. Lappala, L. L. Yaeger, and

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Issued as separate publication *ASTM STP No. 256.*

[2] To be issued as separate publication *ASTM STP No. 273.*

Johan Bjorksten, Bjorksten Research Laboratories.

The Second Edition of the Manual on Industrial Water and Industrial Waste Water (*ASTM STP No. 148-D*) will appear in 1960. Part I will include the following chapters:

I. Uses of Industrial Water
II. Effects of Impurities on Water Uses
III. Self-Purification of Streams[3]
IV. Composition and Classification of Industrial Water and Water-Formed Deposits
V. Treatment of Industrial Water
VI. Sampling and Flow Measurement of Industrial Water
VII. Analysis of Industrial Water
VIII. Sampling and Identification of Water-Formed Deposits
IX. Analysis of Water-Formed Deposits
X. Radioactive Nuclides in Water[3]

Part II will include all tentative and standard methods and specifications prepared by the committee and accepted and published by the Society through June, 1959.

The ASTM representatives to the Joint Committee on Uniformity of Methods of Water Examination (JCUMWE) supplied that committee with recently published new tentatives and revisions of published methods. The representatives made available to Committee D-19 interim JCUMWE Panel Reports on various methods under study. They submitted to the Society official recommendations for the reporting of results of analysis of water, hardness in water, iron in water, and organic nitrogen in water, the first three being submitted in May, the last one in December, 1958.

The Society advised its representatives in July, 1958, that Committee D-19 accepted the JCUMWE recommendations concerning the reporting of results of analysis of water. The representatives were advised in March, 1959, that the committee did not accept the recommendations on the method for hardness in water and submitted reasons therefor. Upon receipt of the ASTM reaction to the recommendation on hardness, JCUMWE voted to amend its recommendation to satisfy the objection. ASTM plans to revise its hardness method to comply with the revised recommendations.

The recommendations relating to iron in water and organic nitrogen in water are being studied by the committee.

The ASTM Award of Merit was presented on June 23, 1959, to C. K. Rice, Coordinator, Refinery Technology Laboratory, Gulf Research and Development Co.

The 1959 Max Hecht Award certificate and citation was presented on June 25, 1959 to L. D. Betz, President, Betz Laboratories, Inc.

The committee was saddened by the loss on May 2, 1958, of one of its active members, L. E. Pitzer, who served as the representative of the United States Steel Corp.

The committee consists of 141 members. The committee continues to operate with one Advisory and six technical subcommittees. As of June 30, 1959, the Society published 33 standards and 33 tentatives prepared by the committee.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Procedure for Determination of Precision of Committee D-19 Methods, as appended hereto.[4]

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[5, 6]

---

[3] This chapter is new material.

[4] The proposed method appears in the Second Edition of the Manual on Industrial Water and Industrial Waste Water (1960).

Tentative Methods of Test for:

Copper in High-Purity Water,
Silica in High-Purity Water,
Measurement of Gamma Radioactivity of Industrial Water and Industrial Waste Water,
Total Chromium in Industrial Water and Industrial Waste Water, and
Zinc in Industrial Water and Industrial Waste Water.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Test for Sulfate Ion in Industrial Water and Industrial Waste Water (D 516 – 55 T):[7] Revise as appended hereto.[5]

Tentative Method of Sampling Steam (D 1066 – 54 T):[7]

Section 6 (b).—Change the word "stream" to "steam" in line 5.

In the last line of Note 2, change "108" to "105."

Change Note 7 to read as follows:

NOTE 7.—When the sample is analyzed for moisture content by throttling or separating calorimeters, the rate of sample flow in the pipe from which the sample is taken is fixed by the characteristics of the calorimeter. The sampling nozzle can be designed for only one rate of flow. See Fig. 4 for the sampling rate for the particular calorimeter orifice at the given steam pressure and Fig. 5 for the steam flow rate in the pipe. Calculate the port area, using the equation given in Section 11 (*c*). See Section 6 (*a*) for the number of ports corresponding to the pipe diameter. Use Fig. 3 to locate the ports in the sampling nozzle. See Figs. 1, 2, 6, 7, and 8 for representative designs.

Change Note 8 to read as follows:

NOTE 8.—When the sample is to be condensed or cooled for analysis, the sampling nozzle may be designed for maximum rate of steam flow and the rate of sample flow may be adjusted for lower rates of steam flow. For the given pipe diameter, determine steam flow at the operating pressure, using Fig. 5. Assume a convenient maximum sample rate consistent with the steam flow and with the equipment provided for condensation and cooling. Calculate the port area as in Note 7.

Figure 6.—In the column heading of the fourth column of the table, change "stream" to "steam." In the last column, opposite Nozzle *B*, change "143" to "112."

Section 7 (b).—Change "ASME Code[6]" to read "American Standard Code for Pressure Piping.[6]"

Footnote 6.—Revise the material within the parentheses to read as follows: "(ASA No. B31.1—1955)."

Section 10 (a).—In Item (*4*) change the word "stream" in line 4 to read "steam."

Section 11 (c).—Revise the equation by enclosing the expression "a/A" in parentheses and eliminating the multiplication sign. Also, change the word "stream" to "steam" in line 5.

Tentative Method of Test for Electrical Conductivity of Industrial Water (D 1125 – 50 T):[7] Revise as appended hereto.[5]

Tentative Scheme for Analysis of Industrial Water and Industrial Waste Water (D 1256 – 53 T):[7] Revise as appended hereto.[5]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Oxidation-Reduction Potential of Industrial Water (D 1498 – 57 T)[7] be approved for reference to letter ballot of the Society for adoption as standard without revision.

---

[5] The new and revised tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 10, and in the Second Edition of the Manual on Industrial Water and Industrial Waste Water.

[6] The results of cooperative tests on these methods are on file at ASTM Headquarters.

[7] 1958 Book of ASTM Standards, Part 10.

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Method of Test for Sulfides in Industrial Waste Water (D 1255 - 55 T):[7]

Title.—Revise to read as follows: "Tentative Method of Test for Sulfides in Industrial Water and Industrial Waste Water."

Section 1 (a).—Before the words "industrial waste water" insert the words "industrial water and."

Section 2.—Insert after "para-amino dimethylaniline" the words "or the para-amino dimethylaniline sulfate."

Section 7 (d).—Add the following words within parentheses and immediately preceding the semicolon in the second sentence: "(or 27.2 g of para-amino dimethylaniline sulfate may be substituted)."

Tentative Method of Test for Evaluating Acute Toxicity of Industrial Waste Water to Fresh-Water Fishes (D 1345 - 54 T):[7]

Note 3.—Change to read as follows:

NOTE.—The concentration of highly volatile and unstable toxicants may decline throughout the test. In the case of highly volatile toxicants, it may be possible to obtain more reliable data by substituting initial oxygenation of the diluent in place of the standard controlled artificial oxygenation procedure. In the case of readily oxidizable constituents, it may be advisable to use test solution renewal techniques.

Section 10.—At the end of Paragraph (*g*) change "(Note 15)" to read "(Notes 15 and 16)."

Following Paragraph (*g*) add the following new note, renumbering subsequent notes and references to them accordingly:

NOTE 16.—In the case of highly volatile test material, it may be advisable to eliminate aeration and to introduce the required amount of oxygen with the dilution water. This can be done by dispersing compressed oxygen gas through the water in a tall cylinder. It is not advisable to use initial oxygen concentrations higher than approximately twice saturation for the test conditions.

Add the following new note at the end of Paragraph (*h*):

NOTE 17.—In the case of highly unstable test material, it may be necessary to renew the test material once every 24 hr or at convenient shorter intervals. This can be accomplished by periodically transferring the test animals quickly by means of a dip-net to a test container with fresh test material. Renewal of the test material at intervals of 24 hr is often both convenient and sufficient, but shorter intervals such as 12 hr or 8 hr sometimes are necessary or advisable. The need for test material renewal can be determined by checking the toxicity of solutions under test for 8 to 24 hr against the toxicity of fresh test solutions.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Definitions of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 - 58),[7] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society.

Revise existing definitions as follows:

Absorbance.—The logarithm to the base 10 of the reciprocal of the relative transmittance, T.

$$A = \log_{10} (1/T) = -\log_{10} T$$

Absorbance thus expresses the excess absorption over that of a specified reference or standard. It is implied that compensation has been effected for reflectance losses, solvent absorption losses, and refractive effects, if present, and that attenuation by scattering is small compared with attenuation by absorption.

Electrical Conductivity.—The reciprocal of the resistance in ohms measured between opposite faces of a centimeter cube of an aqueous solution at a specified temperature.

NOTE.—The electrical conductivity shall be expressed in micromhos per centimeter at t C. The actual resistance, R, of the cell is measured in ohms. The conductance, $1/R$, is directly proportional to the cross-sectional area, A, inversely proportional to the length of the path, L, and directly proportional to the constant, K. The latter is the conductivity measured between opposite faces of a centimeter cube. Mathematically, $\frac{1}{R} = \frac{KA}{L}$, or $K = \frac{L}{AR} = \frac{\text{cm}}{\text{sq cm} \times \text{ohms}}$ = mhos per cm. The numerical value of this expression multiplied by 1,000,000 is the electrical conductivity in micromhos per centimeter.

pH.—A measure of the hydrogen ion concentration of a sample and representing the logarithm to the base 10 of the reciprocal (negative logarithm) of the activity of hydrogen ions, calculated as follows:

$$\text{pH} = \log_{10} (1/\text{H}^+) = -\log_{10} (\text{H}^+)$$

where H^+ = activity of hydrogen ions.

Transmittance.—The ratio of radiant power transmitted by the sample to the radiant power incident on the sample.

NOTE.—In practice, the sample is often a liquid or a gas contained in an absorption cell. In this case, the transmittance is the ratio of the radiant power transmitted by the sample in its cell to the radiant power transmitted by some clearly specified reference material in its cell, when both are measured under the same instrument conditions such as spectral position and slit width. In the case of solids not contained in a cell, the radiant power transmitted by the sample is also measured in the presence of that transmitted by a clearly specified reference material. This ratio is called relative transmittance, T.

New Definitions.—Add the following new definitions in the proper alphabetical sequence:

Operating Cycle.—An ion exchange process consisting of a backwash, regeneration, rinse, and service run.

Phenolic Compounds.—Hydroxy derivatives of benzene and its condensed nuclei.

Rinse.—That part of the operating cycle of an ion-exchange process in which a specified water is passed through the bed of ion-exchange material to remove the residual regenerant solution.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

Acidity and Basicity (Alkalinity) in Industrial Water and Industrial Waste Water (D 1067 – 57 T),

Chloroform-Extractable Matter in Industrial Water and Industrial Waste Water (D 1178 – 54 T), and

Sodium and Potassium Ions in Industrial Water and Water-Formed Deposits by Flame Photometry (D 1428 – 56 T).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following tentatives and standards:

Standard Method of Test for Iron Bacteria in Industrial Water and Water-Formed Deposits (D 932 – 58):[7]

Section 8 (c).—In the first line change the parenthetical acid strength to read "(1:4)."

Standard Method for Examination of Water-Formed Deposits by Chemical Microscopy (D 1245 – 55):[7]

Section 7.—Revise Paragraphs (*c*), (*d*), (*j*), and (*p*) to read, respectively, as follows:

(*c*) *Hydrochloric Acid* (*sp gr 1.19*).—Concentrated hydrochloric acid (HCl).

(*d*) *Hydrochloric Acid* (*1.4*).—Mix 1 volume of HCl (sp gr 1.19) with 4 volumes of water.

(*j*) *Ammonium Molybdate Solution.*—Dissolve 1 g of ammonium molybdate $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 10 ml of water and add 5 drops of HNO_3 (sp gr 1.42).

(*p*) *Chloroplatinic Acid Solution.*—Dissolve 1 g of $H_2PtCl_6 \cdot 6H_2O$ and 5 ml of water and add 0.5 ml of HCl (sp gr 1.19).

Section 10.—In Paragraph (*b*) change the parenthetical strength of HCl to read: "(1:4)."

In Paragraphs (*i*) and (*q*) change the parenthetical specific gravity of HCl to read: "(1.19)."

Standard Methods of Test for Residual Chlorine in Industrial Water (D 1253 - 57):[7]

Footnote 8.—Add the following sentence: "Glassware should be conditioned with water containing at least 10 ppm of residual chlorine for at least 2 hr before use and then rinsed with water having zero chlorine demand."

Standard Methods of Test for Residual Chlorine in Industrial Waste Water (D 1427 - 58):[7]

Footnote 11.—Add the following sentence: "Glassware should be conditioned with water containing at least 10 ppm of residual chlorine for at least 2 hr before use and then rinsed with water having zero chlorine demand."

Tentative Methods of Test for Sodium and Potassium Ions in Industrial Water and Water-Formed Deposits by Flame Photometry (D 1428 - 56 T):[7]

Section 21 (e).—Revise to read as follows: "*Sulfuric Acid (1:4).*—Mix 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) with 4 volumes of water."

Section 23 (*c*).—Change the parenthetical strength of H_2SO_4 to read "(1:4)."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[8]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Definitions and General Specifications (S. K. Love, chairman).—First drafts of the manuscripts have been received for all chapters of the Second Edition of the Manual on Industrial Water and Industrial Waste Water, with the exception of Chapter 5, and editorial revision of the material has been made.

The Task Group on Uniformity of Reagents has reviewed most of the Committee D-19 methods as to concentration of reagents specified and has made recommendations to minimize the number of different reagents required.

The Tentative Scheme for Analysis of Industrial Water and Industrial Waste Water (D 1256) has been revised to include recently adopted methods and outline forms have been used to replace the line drawings showing interrelation of analyses.

A procedure for determination of precision has been prepared for the use of the committee and will ultimately be included in Part III of the Guide.

The subcommittee has approved definitions for absorbance, operating cycle, pH, phenolic compounds, rinse, and transmittance for inclusion in Standard Definitions D 1129.

Subcommittee III on Methods of Sampling of Industrial Water and Flow Measurements (E. F. Davidson, chairman).—The Task Group on Steam Sampling completed the revisions of Method D 1066 referred to earlier in the report.

The Task Group on Sampling Radioactive Industrial Water is studying the need for special methods for sampling. Work by the task group is to continue on a method of sampling heterogeneous industrial water.

The Task Group on Flow Measurement plans to prepare specific methods for flow measurement in open channels by means of Parshall flume and weirs.

Subcommittee IV on Methods of Analysis of Industrial Water (J. F. J. Thomas, chairman).—The subcommittee completed the revision of Method D 516, the proposed Tentative Method of Test for Silica in High-Purity Water, the proposed Tentative Method of Test for Copper in High-Purity Water, and the proposed Tentative Method of Test for Zinc in Industrial Water referred to earlier in the report.

---

[8] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Final drafts were completed and cooperative evaluations are under way on new methods for chloride, copper, and nitrite ions. Progress was made in developing a referee method for sulfite ion and a task group was set up to revise the manganese ion method. Studies were started of changes recommended by JCUMWE on existing methods for hardness and iron.

Subcommittee V on Testing Procedures (A. O. Walker, chairman).—The Task Group on Appearance Properties decided to drop the procedure on color because of lack of interest. Drafts on turbidity and transmittance are in preparation.

The Subcommittee completed the revisions of Method D 1125 referred to earlier in the report.

The Task Group on Flame Photometer has recommended that Method D 1428 be separated into two methods. Method D 1428 will contain procedures for sodium and potassium above 1 ppm and sodium and potassium in deposits. A new method will be prepared for sodium in high-purity water. The scope will cover applicability to water and condensed steam having a sodium content of 1 ppm or less.

The Task Group on Measurement of Radioactivity completed the proposed Tentative Methods of Test for Measurement of Gamma Radioactivity of Industrial Water and Industrial Waste Water referred to earlier in the report.

Subcommittee VI on Corrosivity and Performance Testing (James K. Rice, chairman).—The Task Group on Ion Exchange Performance Testing completed a series of round-robin tests employing the latest draft of their method for cation exchange materials in the sodium cycle. A second set of round-robin tests is contemplated based upon data developed by the first round. A series of tests is to be conducted on a method for cation exchange capacity in the acid cycle.

The Task Group on Tubular Metal Products is considering a method of test employing heat transfer.

The Task Group on Radioactive Tracer Testing has agreed to a condensed list of publications and abstracts that are to be abstracted and referenced by the task group. A division of this work was made among the membership of the group. It is their intention to have an up-to-date bibliography and index available to the membership of the committee by June of this year.

A Task Group on Impurities in Steam was organized with E. E. Coulter as chairman. The task group proposed and had adopted by the committee the following scope: "This method prescribes procedures for determining the amount of liquid entrained in steam from boiling water." It is the intention of the task group to begin work on a draft of a proposed method employing sodium analysis.

A new Task Group on Ion Exchange Properties was formed with Miss Sallie Fisher as chairman. The Task Group has the following scope:

> These methods cover the testing of the physical and chemical properties of ion exchange materials used in the treatment of industrial water. The methods are applicable for the chemical and physical characterization of both anion and cation exchange materials which are to be used or have been used in the treatment of industrial water.

Subcommittee VII on Industrial Waste Water (Robert A. Baker, chairman).—

The joint task groups of Subcommittee VII and Subcommittee IV on Acidity, Basicity (Alkalinity) and pH recommended continuation of Method D 1067 as tentative. Proposed definitions for acidity, basicity, and alkalinity were submitted to the Task Group on Terminology and Nomenclature for consideration.

The Task Group on Chromium reviewed and submitted the tentative Method of Test for Total Chromium in Industrial Water and Industrial Waste Water referred to earlier in the report.

The Task Group on Chlorine has under consideration modifications of Methods D 1291 and D 1427. A special pretreatment has been suggested to minimize error due to chlorine demand of glassware and electrodes.

The Task Group on Cyanides agreed that additional studies of the proposed method are necessary. The amount of cyanide recovered is a function of the time and distillation conditions as well as the nature of the cyanide. A series of tests will be conducted to determine the effect of these variables on complex cyanides.

The Task Group on Dissolved and Suspended Matter has under consideration recent findings on the effect of temperature in determination of total dissolved solids.

A fourth draft of a revision of Method D 1292 was considered by the Task Group on Odor. Suggestions by JCUMWE were appraised to facilitate development of the method and future agreement. Modifications of the method were considered for a fifth draft available at the June meeting. A temperature of 40 C, a possible precision of one dilution, use of 200 ml total volume, development of an "odor dilution index," modification of the system of odor classification, and other points have been tentatively established.

The Task Group on Oily Matter has under review a draft of a proposed method of test for hexane extractable matter.

Several changes, based on JCUMWE recommendations, are anticipated in Method D 1340. These changes include substitution of hexane for benzene as the extraction solvent and associated changes in definition and alternate solvents.

The Task Group on Oxygen Demand is considering a method of test for biochemical oxygen demand (BOD). A method of test for chemical oxygen demand (COD) using silver catalyst on high chloride wastes is being considered and may provide the basis for a future revision of Method D 1252.

The Task Group on Phenolic Compounds reviewed results of round-robin tests at 100 ppb phenol. Precision of ±5 per cent was indicated. Minor changes in draft number 5 of the proposed 4-aminoantipyrine method were agreed upon. Round-robin tests at the 10 ppb and 50 ppb phenol levels are planned.

The Task Group on Reporting Results elected to retain Method D 596 as standard without any changes at this time. However, methods of reporting results for newer methods such as radiation and odor are under consideration. Assignments were made to develop these reporting methods.

The Task Group on Specific Gravity recommended Method D 1429 for retention as tentative with revision. The precision of results from round-robin testing was found satisfactory and these tests will be discontinued.

The Task Group on Sulfur Compounds recommended that Method D 1255 be recommended for adoption as standard with revision. Reagent changes substituting para-amino dimethylaniline sulfate for para-amino dimethylaniline are to be included and the scope enlarged to include industrial water.

The Task Group on Synthetic Surface Active Materials have curtailed development of methods pending the outcome of research on the development of a short colorimetric method for alkylbenzene sulfonate (ABS) in surface water and sewage.

The Task Group on Toxicity to Aquatic Life voted to adopt the Method D 1345 as standard with several minor revisions.

A new method of test for evaluating inhibitory toxicity of industrial wastes to diatoms is nearing completion.

The subcommittee voted to recommend the adoption of Methods D 1255 and D 1345 as standard with revision; to retain Methods D 1067 and D 1178 as tentatives without revision; to retain Method D 1429 as tentative with revision; and to submit as tentative the Method of Test for Total Chromium in Industrial Water referred to earlier in the report.

This report has been submitted to letter ballot of the committee, which consists of 141 members; 92 members returned their ballots, of whom 81 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

O. M. ELLIOTT,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards the following recommendation:

Revision of Tentative Method of Test for:

Odor of Industrial Waste Water (D 1292 – 58 T).

This recommendation was accepted by the Standards Committee on October 2, 1959, and the revised tentative method appears in the Second Edition of the Manual on Industrial Water and Industrial Waste Water (1959).

REPORT OF COMMITTEE D-20
ON
PLASTICS*

The work of Committee D-20 on Plastics moved forward vigorously during the past year as a result of three highly productive meetings: at the Annual Meeting of the Society in Boston, Mass., on June 25, 26, and 27, 1958; at Philadelphia, Pa., on October 27, 28, and 29, 1958; and at Washington, D. C. on February 18, 19, and 20, 1959.

The committee consists of 418 members, of whom 213 are classed as producers, 108 as consumers, and 97 as general interest members. There are 262 voting representatives.

A new subcommittee XXII on Plastics in Tooling was established. A guide for the writing of specifications on plastics was prepared and will appear in the 1959 Compilation of ASTM Standards on Plastics.

The extensive participation of the United States in the work of Technical Committee 61 of the International Organization for Standardization continued through Subcommittee XXI of Committee D-20 which constitutes the U. S. A. Group. The eighth annual meeting of ISO/TC 61 was held in Washington, D. C. in November, 1958. A highly significant symposium on Plastics Testing and Standardization[1] was sponsored by ASTM in Philadelphia in connection with the D-20 meeting just prior to the ISO meeting in Washington.

The first Research Report from Committee D-20 was filed at ASTM Headquarters. It is RR1: D-20 Proposed Method for Classifying PVC Resins.

AMERICAN STANDARDS

The American Standards Association approved as American Standard the following ASTM standards relating to plastics which had been recommended for ASA approval by Committee D-20:

Specifications for:

Ethyl Cellulose Molding Compounds (D 787 - 55; ASA K64.1—1959)

Cellulose Acetate Plastic Sheets (D 786 - 49; ASA K64.2—1959)

Cellulose Nitrate (Pyroxylin) Plastic Sheets, Rods, and Tubes (D 701 - 49; ASA K64.3—1959)

Molds for Test Specimens of Plastic Molding Materials (D 647 - 57; ASA K66.1—1959)

Methods of Test for:

Compressive Properties of Rigid Plastics (D 695 - 54; ASA K64.1—1959)

Stiffness Properties of Nonrigid Plastics as a Function of Temperature by Means of a Torsional Test (D 1043 - 51; ASA K65.2—1959)

Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 - 51; ASA K65.3—1959)

Deformation of Plastics Under Load (D 621 - 51; ASA K65.4—1959)

Haze and Luminous Transmittance of Transparent Plastics (D 1003 - 52; ASA K65.5—1959)

Luminous Reflectance, Transmittance, and Color of Materials (D 791 - 54; ASA K65.6—1959)

Index of Refraction of Transparent Organic Plastics (D 542 - 50; ASA K65.7—1959)

Specific Gravity of Plastics (D 792 - 50; ASA K65.8—1959)

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

[1] Issued as separate publication *ASTM STP No. 247*.

Total Chlorine in Vinyl Chloride Polymers and Copolymers (D 1303 – 55; ASA K65.9—1959)
Dilute Solution Viscosity of Vinyl Chloride Polymers (D 1243 – 58 T; ASA K65.10—1959)
Ammonia in Phenol-Formaldehyde Molded Materials (D 834 – 57; ASA K65.11—1959)
Acetone Extraction of Phenolic Molded or Laminated Products (D 494 – 46; ASA K65.12—1959)
Apparent Density and Bulk Factor of Granular Thermoplastic Molding Powder (D 1182 – 54; ASA K66.2—1959)

Methods of Measuring:

Shrinkage from Mold Dimensions of Molded Plastics (D 955 – 51; ASA K66.3—1959)

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the recommendations listed below. The recommendations were accepted by the administrative committee on the dates indicated. The new Tentative Specifications D 1527 and D 1528 appear in the 1958 Book of ASTM Standards, Part 9; the other recommendations will appear in the 1959 Supplement to Part 9.

New Tentative Method of Test for:

Compressive Strength of Rigid Cellular Materials (D 1621 – 59 T) (Approved March 25, 1958),
Tensile Properties of Rigid Cellular Materials (D 1623 – 59 T) (Approved March 25, 1959),
Apparent Density of Rigid Cellular Materials (D 1622 – 59 T) (Approved March 25, 1959),
Tensile Heat Distortion Temperature of Plastic Sheeting (D 1637 – 59 T) (Approved March 25, 1959), and
Chemical Analysis of Urethane Foam Raw Materials (D 1638 – 59 T) (Approved July 13, 1959).

New Tentative Specifications for:

Extruded Acrylic Plastic Sheet (D 1547 – 59 T) (Approved March 25, 1959).
Allyl Molding Compounds (D 1636 – 59 T) (Approved March 25, 1959),
Dimensions of Iron Pipe Size (IPS) Extruded Acrylonitrile-Butadiene-Styrene (ABS) Plastic Pipe (D 1527 – 58 T) (Approved Sept. 9, 1958),
Dimensions of Solvent Welded (SWP Size) Extruded Acrylonitrile-Butadiene-Styrene (ABS) Plastic Pipe (D 1528 – 58 T) (Approved Sept. 9, 1958), and
Cellulose Propionate Molding and Extrusion Compounds (D 1562 – 58 T) (Approved Sept. 9, 1958).

New Tentative Recommended Practice for:

Operating Light- and Water-Exposure Apparatus (Carbon Arc Type) for Exposure of Plastics (D 1499 – 59 T) (Approved March 25, 1959).

Revision of Tentative Specifications for:

Polyester Molding Compounds (D 1201 – 58 T) (Approved March 25, 1959),
Cast Methacrylate Plastic Sheets, Rods, Tubes, and Shapes (D 702 – 58 T) (Approved March 25, 1959), and
Nylon Injection Molding and Extrusion Material (D 789 – 53 T) (Approved March 25, 1959).

Revision of Tentative Method of Test for:

Flexural Properties of Plastics (D 790 – 58 T) (Approved March 25, 1959).

Revision of Tentative Definitions of:

Terms Relating to Plastics (D 883 – 58 T) (Approved March 25, 1959).

Tentative Revision of Standard Method of Test for:

Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 58) (Approved July 13, 1959).

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications, methods of test, and recommended practice as appended hereto:[2]

Tentative Method of:

Measuring Absorbed Gamma Radiation Dose by Fricke Dosimetry (Joint with D-9),
Test for Flammability of Plastic Foams and Sheeting, and
Test for Environmental Stress Cracking of Type I Ethylene Plastics.

Recommended Practice for:

Exposure of Polymeric Materials to High Energy Radiation (Joint with D-9).

---

[2] The new and revised tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 9.

Tentative Specifications for:

Thermosetting Reinforced Plastic Pipe Thread System.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Water Absorption of Plastics (D 570 - 57 T):[3]

Section 6.—At the end of Paragraph (*b*), change "2 hr" to read "120 ± 4 min."

Revise Paragraph (*d*) to read as follows:

> (*d*) *Long-Term Immersion.*—To determine the total water absorbed when substantially saturated, the conditioned specimens shall be tested as described in Paragraph (*a*) except that at the end of 24 hr they shall be removed from the water, wiped free of surface moisture with a dry cloth, weighed immediately, and then replaced in the water. The weighings shall be repeated at the end of the first week and every two weeks thereafter until the increase in weight per two week period, as shown by three consecutive weighings, averages less than 1 per cent of the total increase in weight, or 5 mg, whichever is greater; the specimen shall then be considered substantially saturated. The difference between the substantially saturated weight and the dry weight shall be considered as the water absorbed when substantially saturated.

New Paragraphs.—Add the following new Paragraphs (*e*), (*f*) and (*g*), relettering the present Paragraph (*i*) as (*h*):

> (*e*) *2-hr Boiling Water Immersion.*—The conditioned specimens shall be placed in a container of boiling distilled water, and shall be supported on edge and be entirely immersed. At the end of 120 ± 4 min, the specimens shall be removed from the water and cooled in distilled water maintained at room temperature. After 15 ± 1 min, the specimens shall be removed from the water, one at a time, all surface water removed with a dry cloth, and the specimens weighed immediately. If the specimen is $\frac{1}{16}$ in. or less in thickness, it shall be weighed in a weighing bottle.
>
> (*f*) *½-hr Boiling Water Immersion.*—For all thicknesses of materials having a relatively high rate of absorption, and for thin specimens of other materials which may show a significant weight increase in ½ hr, the specimens shall be tested as described in Paragraph (*e*), except that the time of immersion shall be reduced to 30 ± 1 min.
>
> (*g*) *Immersion at 50 C.*—The conditioned specimens shall be tested as described in Paragraph (*e*), except that the time and temperature of immersion shall be 48 ± 2 hr and 50 ± 1 C (122.0 ± 1.8 F), respectively, and cooling in water before weighing shall be omitted.

Section 8.—Add a new Item (*3*) to read as follows, renumbering subsequent items accordingly: "(*3*) Immersion procedure used." Revise the present Item (*3*) to read "(*4*) Time of immersion (Procedure (*d*) only)." Revise the present Item (*6*) to read "(*7*) The percentage of water absorbed, which is the sum of the values in Items (*5*) and (*6*), and."

Tentative Method of Test for Tear Resistance of Plastic Film and Sheeting (D 1004 - 49 T):[3] Revise as appended hereto.[2]

Tentative Method of Test for Dilute Solution Viscosity of Ethylene Polymers (D 1601 - 58 T):[3] Revise as appended hereto.[2]

TENTATIVE REVISIONS OF STANDARD

Committee D-20, jointly with Committee D-9 on Electrical Insulating Materials, recommends tentative revisions as appended hereto[4] of the Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 - 58).[3]

The proposed revision is intended to provide a convenient uniform means of describing, in an abbreviated form, standard procedures for conditioning and environments for testing. Similar systems of nomenclature, from which the proposed one is derived, are described in Section 12 of the Tentative Specifications for Laminated Thermosetting Materials (D 709 - 55 T) and in Armed Forces Specifications MIL-P designation, and are used extensively in the industry. The proposed compromise is intended to lessen confusion (for example, between Procedure A of Methods D 618 and Condition A of Specifications D 709) and to eliminate the need for cross reference or lengthy explanations.

---

[3] 1958 Book of ASTM Standards, Part 9.

[4] The tentative revision appears in the 1959 Supplement to Book of ASTM Standards, Part 9.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Test for Measuring the Flow Properties of Thermoplastic Molding Materials (D 569 - 48):[3]

Section 3 (a).—At the beginning of the first sentence omit the words "A vertieal orifice $\frac{1}{8}$ in. in diameter" and substitute the following: "A vertical orifice 0.125 ± 0.0005 in. in diameter."

Standard Methods of Test for Deformation of Plastics Under Load (D 621 - 51):[3]

Section 5(a).—Add the following sentence at the end of the paragraph: "Surfaces of the test specimens shall be plane and parallel."

New Note.—Add the following new Note at the end of Section 5:

NOTE.—Substantial variations in test values may occur, particularly with composite specimens, if the opposite faces of the specimens are not plane and parallel or if sink marks or other similar imperfections are present. To minimize such variations, the squares used to form the composite specimens, if necessary, shall be rendered plane and parallel and their adjoining surfaces shall be freed of sink marks and other imperfections by milling, grinding, or other appropriate means. If solid cubes are used, their opposite faces in contact with the anvils of the testing machine, if necessary, shall be milled plane and parallel and shall be smooth and free of imperfections.

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the Standard Specifications for Cast Allyl Plastic Sheets, Rods, Tubes, and Shapes (**D 819 - 50**).[3]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Test for:

Acetone Extraction of Phenolic Molded or Laminated Products (**D 494 - 46**), and

Bond Strength of Plastics and Electrical Insulating Materials (**D 952 - 51**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Properties (P. Z. Adelstein, chairman).—Revision of the Tentative Method of Test for Tear Resistance of Plastic Film and Sheeting (D 1004 - 49 T) has been completed.[2] Work almost completed includes the test for indentation hardness of plastics (Shore A and D), and the revisions to the tensile definitions appended to Method D 638. A round robin has been completed on the microtension test and a draft of a method has been proposed. Two additional round-robin progıams were completed on the tensile impact test which resulted in a working draft of a method. Round-robin testing programs are currently under way on measurement of blocking resistance and friction and the use of the torsion pendulum. The test methods for Compressive Properties of Rigid Plastics (D 695 - 54), the Method of Test for Repeated Flexural Stress (Fatigue) of Plastics (D 761 - 51 T), and the Elmendorf tear test are now being revised.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Effects of Nuclear and High Energy Radiation (D. S. Ballantine, chairman), a joint subcommittee with Committee D-9, is recommending adoption of the "rad" as the unit to be used in expressing absorbed radiation doses. The Section on Dosimetry has tested and approved a ferrous-sulfate chemical dosimeter for use in measuring total doses up to 40,000 rads, incorporated in the proposed Tentative Method of Measuring Absorbed Gamma Radiation Dose by Fricke Dosimetry.[2] The Recommended Practice for Exposure of Polymeric Materials to High Energy Radiation[2] has been approved for committee ballot. Three lectures covering the basic considerations of radiation chemistry have been given at joint meetings of Committees D-9 and D-20.

A symposium on Postirradiation Effects in Polymers[6] is being arranged in connection with the October, 1959, Pacific Area National Meeting of the Society in San Francisco.

Subcommittee III on Thermal Properties (A. A. Harban, chairman).—The section which was formed last year to develop a Tentative Method of Test for Tensile Heat Distortion Temperature of Plastic Sheeting (D 1637 - 59 T) has already completed its work, and the new method has been accepted by the Administrative Committee on Standards as stated earlier in the report. The flammability section has developed a proposed method of test for measuring the surface flammability of plastics using a radiant heat energy source and has submitted it to Committee E-5 for consideration as a method applicable to other materials. It is also recommending a proposed Method of Test for Flammability of Plastic Foams and Sheeting.[2] Method D 746 for measuring the brittleness temperature of plastics was revised to correct an error in test specimen configuration, and a more drastic revision is in progress to improve the utility of the method as applied to crystalline polymers such as polyethylene. This revision will involve the use of a notched specimen. A round-robin on the SPI test for low temperature brittleness of plastic films was completed, and a tentative procedure is being drafted. A recommended practice for presentation of capillary flow data on molten thermoplastics has been submitted to Committee D-20, and revision of the Test for Measuring Flow Rates of Thermoplastics (D 1238) is in progress. Minor revisions in Methods D 569 and D 621 were prepared, as noted earlier in the report. A new section on ablation tests was formed to develop methods of characterizing the behavior of materials at the very high temperatures encountered in the rocket and missile fields. A symposium on Thermal Ablation will be held at the Third Pacific Area National Meeting of the Society in San Francisco the week of October 11, 1959.[6]

Subcommittee IV on Optical Properties (D. A. Popielski, chairman).—An objective test method, based on specular transmittance, has been developed for describing the optical clarity property (see-through distinctiveness of detail) of plastic films, but additional work aimed at ascertaining optimum instrument geometry for the measurement of specular transmittance over the narrow receptor angles required is necessary.

---

[6] To be issued as separate publication *ASTM STP No. 276.*

Work on the development of an instrumental method for the measurement of yellowness of transparent plastic materials is essentially complete; plans have been formalized to develop a similar test method applicable to pigmented opaque plastics.

In accordance with the preferred terminology adopted at the 1951 meeting of the Commission Internationale de l'Éclairage, the subcommittee voted to change the term "illuminant" to read "source" in all methods under its jurisdiction.

Subcommittee V on Permanence Properties (B. G. Achhammer, chairman) is writing a proposed tentative recommended practice for use of dyed acrylic plastic for comparing relative rates of exposure to radiation in light- and water-exposure apparatus (carbon-arc type). The round-robin data on which this method is based are being assayed for proposed publication in the ASTM BULLETIN. A Recommended Practice for Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Exposure of Plastics (D 1499 – 59 T) was accepted by the Administrative Committee on Standards. This recommended practice was developed to incorporate Recommended Practice E 42 for general use of light- and water-exposure apparatus (carbon-arc type) in a method designed for plastics.

An alternate procedure for Method D 1203 involving the use of a wire cage around the test specimen as in the ISO procedure is being developed. A revision of Recommended Practice D 794 to extend the heating time to 550 hr is under way. A revision of Method D 570, to change the end point of the long-term immersion and to obtain conformity with some parts of the ISO/TC 61 method, is set forth earlier in the report. A revision of Specifications D 543 is being balloted upon.

Subcommittee VII on General Analytical Methods (J. B. DeCoste, chairman) has been reorganized with the sections classified according to the analysis technique employed: Section A, on General Physical Properties; Section H, on Spectroscopic Methods; and Section K on Chemical Methods. Active projects on physical properties include the compilation of the bulk density Methods D 392, D 954, and D 1182 into a single standard; and the revision of Methods D 792 and D 1505. A revision of Method D 834 to include an alternate qualitative procedure for the determination of ammonia in phenolic plastics was approved for ballot by Committee D-20. The jurisdiction for the development of specific test methods on poly(vinyl chloride) resins for incorporation in a specification has been transferred to Section XV-H on Vinyl Resins.

Subcommittee VIII on Research (A. G. H. Dietz, chairman) has continued to arrange for presentation of papers[7] on subjects of current interest to the committee.

At the fall meeting two papers were presented:

> "Program for the Development of Reinforced Plastics Engineering Design Manuals," by John H. Gibbud of Owens-Corning Fiberglas Corp.
>
> "Equipment for Characterization of Melt Behavior of Thermoplastics," by R. R. Cosner of Union Carbide Chemicals Co.

At the spring meeting the program was as follows:

> "Time and Temperature Dependence of Small Deformation and Ultimate Properties of Amorphous Polymers Above the Glass Tem-

---

[7] There are no plans for publication of these papers by the Society although individual papers may have been submitted. Authors can supply information.

perature," by Thor L. Smith, Jet Propulsion Laboratory, California Institute of Technology.

"The Measurement of the Brittleness Temperature of Polyethylene," by A. Rudin and A. M. Birks of Canadian Industries, Ltd.

"Laboratory Tests as an Indication of End-Item Suitability," by George R. Rugger of Picatinny Arsenal.

Subcommittee IX on Molds and Molding (G. H. Williams, Jr., chairman).—Sections B and D have jointly completed the round robin for molding phenolic test specimens and the data are now being analyzed statistically. It was agreed to publish the findings of the round robin. Section B will study the conclusion for revision of Recommended Practice D 796, and Section D will prepare recommended practices for transfer molding of phenolic test specimens. Section H has completed its round robin on a proposed standard injection molding procedure for type III polystyrene with very encouraging results. Certain desirable changes were indicated and a second somewhat modified round robin was agreed upon. The miniature injection molding machine program in Section H has progressed to the extent that five participating laboratories have agreed to build molds and work out the final details. Section J task group is now considering three possible methods of measuring mold release. The objective will be to select a suitable die and test method.

Subcommittee X on Definitions, Nomenclature and Significance Tests (P. E. Willard, chairman).—A program has been initiated to compile all definitions under the jurisdiction of Committee D-20 into Definitions D 883. It is hoped that this compilation will serve as the basis for a dictionary of plastic terms. The subcommittee reviewed the ISO recommendation for approximately 800 equivalent terms in English, French, and Russian in order to present the American viewpoint on this document. Work has continued on the development of definitions of terms relating to plastics.

Subcommittee XI on Editorial Review (G. M. Kline, chairman) has reviewed, and in some instances revised editorially, the proposed standards and revisions prepared by the subcommittees of Committee D-20.

Subcommittee XIII on Statistical Techniques (R. H. Supnik, chairman) was formed to deal with problems of sampling and design of interlaboratory studies peculiar to the activities of Committee D-20. Two sections have been formed to draw up recommended practices and preliminary work is under way.

A series of indoctrination lectures has been planned to acquaint Committee D-20 members with statistical theory and applications and to provide some background for using statistical concepts. The first of these, held during the February, 1959, meeting in Washington with W. J. Youden of the National Bureau of Standards as the guest speaker, was extremely well attended and received.

Subcommittee XIV on Conditioning (G. M. Armstrong, chairman) has prepared a tentative revision of Standard Methods of Conditioning Plastics and Electrical Insulating Materials (D 618 – 58)[3] which provides a more specific and a more realistic statement of temperature and relative humidity tolerances for the standard laboratory atmosphere. It provides for actual measurement of temperature and humidity ranges in the location used for testing or preconditioning, rather than reliance on indicators or controllers elsewhere to determine the ranges. Provisions of this revision are in essential agreement with those of the international methods accepted by ISO/TC-61 and IEC/TC-15.

The subcommittee has also prepared a proposed further tentative revision of this standard which provides a convenient abbreviated nomenclature for describing conditioning procedures, as presented with this report. Further study of the significance and requirements for temperature and relative humidity tolerances is in progress. The work of this subcommittee is a joint activity of Committees D-9 and D-20.

Subcommittee XV on Thermoplastic Materials (L. Gilman, chairman)[5] established during the year a Section P on Vinyl Dispersions which functions as a joint committee with the Society of the Plastics Industry, in particular the Vinyl Dispersions Division. Work proceeded on the development of new specifications for rigid poly(vinyl chloride) compounds, styrene-butadiene molding and extrusion materials, polytetrafluoroethylene rod, and rigid ABS plastics. Work proceeded on the revision of several existing specifications as well as on a number of test methods for thermoplastic materials. The name of Section J was changed from "ethylene plastics" to "polyolefins."

Subcommittee XVI on Thermosetting Materials (J. E. Hanna, chairman) prepared the new Tentative Specification for Allyl Molding Compounds (D 1636 – 59 T), and the revision of Specification D 1201, for Polyester Molding Compounds, covering the addition of Type 6. Section E on Casting Materials has prepared a specification on epoxy casting resins which is ready for subcommittee ballot. Work on the revision of D 704 – 56 T, Specification for Melamine-Formaldehyde Molding Compounds, is in hand.

Subcommittee XVII on Plastic Pipe and Fittings (George H. Reed, chairman).—The work of this subcommittee resulted in the approval during the year of several important test methods and specifications for plastic pipe. Among these are Dimensions of Iron Pipe Size (IPS) Extruded Acrylonitrile-Butadiene-Styrene (ABS) Plastic Pipe (D 1527 – 58 T) and Dimensions of Solvent Welded (SWP Size) Extruded Acrylonitrile-Butadiene-Styrene (ABS) Plastic Pipe (D 1528 – 58 T).

Subcommittee XVIII on Reinforced Plastics (H. A. Perry, chairman) pursued methods and specification activities during the year on flat sheets, pipe, corrugated panels, and high-temperature resistant materials. A significant and successful effort was made to integrate the activities of subcommittee members from the West Coast with those on the East Coast. The subcommittee has arranged a symposium on Reinforced Plastics[6] at the Third Pacific Area National Meeting of the Society in San Francisco the week of October 11, 1959.

Subcommittee XIX on Plastic Film and Sheeting (K. A. Kaufmann, chairman).—A specification for rigid vinyl chloride sheeting is being worked on as well as a test method for determining welding performance of poly(vinyl chloride) structures. Several test methods for polyethylene film are in various stages of development and work on a polyethylene film specification is temporarily delayed pending agreement on these methods. Work is proceeding on development of thickness tolerances to cover the recent inclusion of a grade 3 in the Specifications D 702.

Subcommittee XX on Cellular Plastics (Samuel Steingiser, chairman) prepared the new Tentative Methods of Test for Compression, Tension, and Density of Rigid Cellular Plastics and Methods of Test for Chemical Analysis of Raw Materials for Urethane Foams which were accepted during the year by the Administrative Committee on Standards.

Section A on Test Methods is actively engaged in completing methods on open and closed cell content, volume determination, and five methods on vinyl raw materials. Liaison has continued with Committee D-9 on such electrical tests as dielectric constant and loss factor of foams. Work at the round-robin stage is in progress on water absorption and on thermal conductivity using these different methods. New work will begin on flexure, moisture vapor transmission, dimensional stability, and additional flammability tests. Work will begin on expandable beads, porosity, etc.

Section B on Urethanes will write a general specification for these materials. Section E on Polystyrene has completed a material specification on expanded foam, and Section F on Nomenclature has approved a number of important definitions which will be submitted to Subcommittee X for review and acceptance by the full committee.

Subcommittee XX is grateful to the Cellular Plastics Division of the Society of the Plastics Industry for its assistance in the initial development of the test methods presently completed with a consequent significant saving of time and effort for ASTM.

Subcommittee XXI, USA National Committee for ISO/TC 61 on Plastics (W. E. Brown, chairman).—The United States continued to participate extensively in the work of Technical Committee 61 of the International Organization for Standardization through Subcommittee XXI of Committee D-20. Fifty-six delegates from twelve countries including sixteen delegates from the United States attended the Eighth Annual Meeting of ISO/TC 61 at Washington, D. C., November 3 through 8, 1958.

Several delegates participated prior to the TC 61 meeting in a plastics testing and standardization symposium under chairmanship of A. C. Webber, E. I. du Pont de Nemours and Co. A summary of this highly significant symposium appeared in the ASTM BULLETIN December, 1958. Full proceedings will be published by the Society as *ASTM Special Technical Publication No. 247*.

The eight Working Groups of ISO/TC 61 held fourteen sessions and treated thirty-five items on the program of work including five new Draft ISO Recommendations on standard atmospheres for conditioning and testing, melt flow index of polyethylene and its compounds, and recommended practices for compression molding specimens of thermoplastics, compression molding specimens of thermosets and injection molding specimens of thermoplastics. Twenty-nine methods have been promulgated by ISO/TC 61 as Draft ISO Recommendations since the committee's inception.

Five of the above-mentioned recommendations have been accepted by the ISO Council and approved as ISO Recommendations while eight others have been sent to the General Secretariat for further action toward adoption as ISO Recommendations. The Draft ISO Recommendation of nearly 800 equivalent terms in English, French, and Russian was revised and will likewise be submitted to the General Secretariat for approval and publication.

Six new Draft ISO proposals were approved for letter ballot including tensile properties of plastics, testing plastics with the torsion pendulum, viscosity number of polyamide resins in solution, acetone soluble matter of phenolic molding materials, tracking under moist conditions, and Vicat softening point.

A new working group on specifications for identification and quality control is being organized by the U.S.A. Group through the American Standards Association. Plans were laid to establish liaison with ISO/TC 45 on Rubber regarding standardization activities on cellular materials. Resolutions were also adopted requesting ISO/ATCO to prepare a document on measurement and control of relative humidity in large and small enclosures, and requesting the secretariat to arrange liaison with ISO/METESCO, the Coordinating Committee on Mechanical Testing of Metals.

A number of reports on the committee's annual meeting have appeared in various technical and trade journals, including the December 1958 ASTM BULLETIN.

The 1959 meeting of ISO/TC 61 will be held in Munich, Germany, October 26–31 following an International Plastics Exhibition in Düsseldorf.

W. E. Brown of the Dow Chemical Co. replaced C. H. Adams of Monsanto Chemical Co. as chairman of the U.S.A. Group following Mr. Adams' resignation.

This report has been submitted to letter ballot of the committee, which consists of 268 voting members; 143 members returned their ballots, of whom 112 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. W. REINHART,
Chairman.

R. M. BERG,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the recommendations listed below. The recommendations were accepted by the Standards Committee on the dates indicated. The new Tentative Methods D 1708 and D 1709, and the revised Tentative Method D 570 are available as separate reprints; the other recommendations appear in the 1959 Supplement to the Book of ASTM Standards, Part 9.

New Tentative Method for:

Particle Size Analysis of Powdered Polymers and Copolymers of Vinyl Chloride (D 1705 – 59 T) (Accepted Oct. 27, 1959).

New Tentative Method of Test for:

Indentation Hardness of Plastics by Means of a Durometer (D 1706 – 59 T) (Accepted Nov. 23, 1959),

Tensile Properties of Plastics: Microtensile Specimens (D 1708 – 59 T) (Accepted Dec. 28, 1959), and

Impact Resistance of Polyethylene Film by the Free Falling Dart Method (D 1709 – 59 T) (Accepted Dec. 28, 1959).

New Tentative Recommended Practice for:

Presentation of Capillary Flow Data on Molten Thermoplastics (D 1703 – 59 T) (Accepted Oct. 27, 1959).

Revision of Tentative Specification for:

Polyethylene Molding and Extrusion Materials (D 1248 – 58 T) (Accepted Nov. 6, 1959).

Revision of Tentative Method of Test for:

Water Absorption of Plastics (D 570 – 59 T) (Accepted Dec. 28, 1959).

Revision and Reversion to Tentative of Standard Method of Test for:

Ammonia in Phenol-Formaldehyde Molded Materials (D 834 – 57) (Accepted Nov. 6, 1959), and

Haze and Luminous Transmittance of Transparent Plastics (D 1003 – 52) (Accepted Oct. 27, 1959).

REPORT OF COMMITTEE D-21
ON
WAX POLISHES AND RELATED MATERIALS*

Committee D-21 on Wax Polishes and Related Materials held two meetings during the year: in New York, N. Y., on December 11, 1958, and in Chicago, Ill., on May 21, 1959.

Additions and changes during the year brought the membership of the committee to 67, of whom 32 are classified as producers, 12 as consumers, 23 as general interest members, and one consulting member.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test For:

Acid Number (Empirical) of Natural Waxes (**D 1386 – 55 T**),[1] and

Saponification Number (Empirical) of Natural Waxes (**D 1387 – 55 T**).[1]

TENTATIVES CONTINUED WITHOUT REVISION

The subcommittees of Committee D-21 have reviewed existing methods of test which have been published by the Society for two years or more, and the committee recommends that the following be continued as tentative without revision:

Tentative Method of Test For:

Nonvolatile Matter (Total Solids) of Water-Emulsion Waxes (**D 1289 – 55 T**),

Application of Emulsion Floor Polishes to Substrates for Testing Purposes (**D 1436 – 56 T**),

Flash Point of Solvent-Type Liquid Waxes (**D 1437 – 56 T**), and

60-deg Specular Gloss of Emulsion Floor Polish (**D 1455 – 56 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (J. T. Hohnstine, chairman) is continuing work on a glossary of terms relating to wax polishes.

Subcommittee II on Raw Materials (Melvin Fuld, chairman) recommended the adoption as standard of Tentative Methods D 1386 – 55 T and D 1387 – 55 T for the acid number and saponification number of natural waxes. Task groups are preparing additional test methods for various properties of natural waxes.

Subcommittee III on Physical and Chemical Testing (Gerald DeNapoli, chairman) is continuing work on a test method for the stability of water emulsion waxes and a method for the determination of nonvolatile matter in solvent waxes.

A task group is investigating methods

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] 1958 Book of ASTM Standards, Part 10.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

for the determination of silicones in wax polishes.

Subcommittee IV on Performance Tests (C. S. Kimball, chairman) prepared a report on "Evaluating the Slip-Resistance of Floor Waxes" which appeared in the September, 1958, ASTM BULLETIN. A task group is investigating the interrelationship between static and dynamic coefficients of friction.

Work on methods for testing the water-spotting and removability of floor waxes is nearing completion.

A task group is developing methods for testing the performance of solvent waxes. One project covering the measurement of thickness of wax films and their wearing properties is well along toward completion.

Another task group is also investigating methods of determining the soil resistance of water emulsion waxes.

Subcommittee V on Specifications (Wm. Joy, chairman) has nearly completed a specification for industrial-type water emulsion floor waxes. Completion of this project awaits the development by the other subcommittees of test methods for some of the properties involved.

This report has been submitted to letter ballot of the committee, which consists of 71 members; 46 members have returned their ballots, of whom 41 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. W. WALTON,
Chairman.

BAYARD S. JOHNSON,
Secretary.

REPORT OF COMMITTEE D-23
ON
CELLULOSE AND CELLULOSE DERIVATIVES*

Committee D-23 on Cellulose and Cellulose Derivatives held one meeting during the year: in Chicago, Ill., on September 8, 1958. The Executive Subcommittee also met in New York City on February 19, 1958.

The committee consists of 48 members, of whom 16 are classified as producers, 11 as consumers, and 21 as general interest members.

NEW TENTATIVES

The committee recommends for publication as tentative the following definitions and method of test as appended hereto:[1]

Tentative Method of Test for:

Solubility of Cellulose in Sodium Hydroxide.

Tentative Definitions of:

Terms Relating to Cellulose and Cellulose Derivatives.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Pentosans in Cellulose (Aniline Acetate Colorimetric Method) (**D 1438 – 56 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (Kyle Ward, Jr., chairman) has prepared the proposed Definitions of Terms Relating to Cellulose and Cellulose Derivatives referred to earlier in the report.

Subcommittee II on Cellulose (E. S. McColley, chairman) has prepared the proposed Tentative Method of Test for Solubility of Cellulose in Sodium Hydroxide, as referred to earlier in this report.

The Task Group on Disperse Viscosity has prepared a procedure which is being used for interlaboratory testing. The Task Group on Pentosans has conducted interlaboratory testing of three methods for pentosans; the Swedish orcinol procedure appeared to be the best. The Task Group on Absorption and Color is engaged in preparing methods for these measurements.

Subcommittee III on Organic Esters (L. B. Genung, chairman) has sent out procedures for acetyl (alternate), hydroxyl, intrinsic viscosity, and color and haze, for consideration by the members. The selected procedures are to be included in Method D 871.

Subcommittee IV on Inorganic Esters (C. B. Gilbert, chairman) has submitted to Committee D-1 on Paint, Varnish, Lacquer, and Related Products the single method for the determination of dilution ratios of cellulose nitrate, solvents, and diluents. Comments received on the method are being reconciled.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new tentatives appear in the 1959 Supplement to Book of ASTM Standards, Part 6.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Cellulose Ethers (R. W. Swinehart, chairman).—The Task Group on Purified Sodium Carboxymethylcellulose is engaged in interlaboratory work on this material. Also, methods for hydroxyethyl cellulose are being prepared by the Task Group on Hydroxyalkyl Ethers.

Subcommittee VI on Statistics (A. F. Johnson, chairman).—Copies of various portions of the manual on statistical methods have been distributed during the year.

This report has been submitted to letter ballot of the committee, which consists of 48 voting members; 25 members returned their ballots, of whom 24 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. A. SIMMONDS,
Chairman.

W. W. BECKER,
Secretary.

REPORT OF COMMITTEE D-24
ON
CARBON BLACK*

Committee D-24 on Carbon Black held two meetings during the year: in Boston, Mass., on June 26, 1958, and in Pittsburgh, Pa., on February 4, 1959.

J. E. Smith of J. M. Huber Corp. has succeeded J. F. Ambrose as chairman of Subcommittee III on Optical Tests. T. D. Bolt of Godfrey L. Cabot, Inc. was appointed as Secretary of Committee D-24 for the remainder of the term to replace A. G. Cobbe who has resigned.

The committee consists of 43 members, of whom 10 are classified as producers, 26 as consumers, and 7 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee D-24 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of Test for:

Discoloration of Benzene by Carbon Black (**D 1618 - 58 T**),
Sulfur Content of Carbon Black (**D 1619 - 58 T**), and
Volatile Content of Carbon Black (**D 1620 - 58 T**).

The recommendations were accepted by the Standards Committee on September 9, 1958, and the new tentatives appear in the 1958 Book of ASTM Standards, Part 9.

The new Tentative Method of Test for Volatile Content of Carbon Black provides a procedure for determining the amount of volatile constituents of carbon black liberated by heating at a specified temperature for a specified period of time. The new Tentative Method of Test for Sulfur Content of Carbon Black provides two procedures for determining the total sulfur present in a carbon black.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Ash Content of Carbon Black (**D 1506 - 57 T**),[1] and
Heating Loss of Carbon Black (**D 1509 - 57 T**).[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Physical Tests (J. F. Svetlik, chairman) is working on a new test method for mass strength of pelleted carbon black.

Subcommittee II on Chemical Tests (L.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] 1958 Book of ASTM Standards, Part 9.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

G. Mason, chairman) prepared the Methods of Test for Volatile Content and for Sulfur Content of Carbon Black referred to earlier in the report.

Subcommittee III on Optical Tests (J. E. Smith, chairman) prepared the Method of Test for Discoloration of Benzene by Carbon Black referred to earlier in the report. Action on color and relative tinting strength tests was deferred.

Subcommittee V on Sampling (R. O. Treat, chairman) will prepare procedures for sampling bag shipments of carbon black and is continuing work on methods for sampling bulk shipments.

This report has been submitted to letter ballot of the committee, which consists of 43 members; 39 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

N. P. BEKEMA,
Chairman.

T. D. BOLT,
Secretary.

REPORT OF COMMITTEE D-25
ON
CASEIN AND SIMILAR PROTEIN MATERIALS*

Committee D-25 on Casein and Similar Protein Materials held one meeting during the year: on February 5, 1959, in Pittsburgh, Pa.

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Sampling Casein and Similar Protein Materials as appended hereto.[1]

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Editorial and Nomenclature (M. E. Stonebreaker, chairman) has approved proposed tests for moisture content and nitrogen. Test methods for oil, ash, fat, free acidity, and alkali requirement are being reviewed.

Subcommittee II on Sampling (J. C. Rice, chairman) has submitted the proposed Method of Sampling Casein and Similar Protein Materials appended to this report.

Subcommittee III on Chemical Tests (J. S. Baker, chairman).—Mr. Baker has resigned as chairman of the subcommittee and Mr. R. A. Olson of the Central Soya Processing Co. has been appointed to take his place. Test methods for ash, free acidity, fat, and alkali requirement are being developed.

Subcommittee IV on Physical Tests (E. M. Maynard, chairman).—Proposed methods on physical tests are presently being evaluated in a series of interlaboratory tests. These tests include particle size, viscosity, insolubles, dirt content, foaming, odor, minimum alkali requirement, and pigment binding properties. Samples of casein and soy protein were sent out in January to the participating laboratories.

Subcommittee VI on Research (L. E. Clark, Jr., chairman).—Various research projects are being reviewed, but none has yet been adopted.

This report has been submitted to letter ballot of the committee which consists of 23 members; 14 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. W. SHADER,
Chairman.

L. E. GEORGEVITS,
Secretary.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 6.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-26
ON
HALOGENATED ORGANIC SOLVENTS*

Committee D-26 on Halogenated Organic Solvents held its organizational meeting at Boston, Mass., during the 1958 Annual Meeting of the Society; and a meeting at Pittsburgh, Pa., during ASTM Committee Week.

The officers elected for the ensuing term of two years are as follows:

Chairman, V. E. Amspacher.

Vice-Chairman, F. G. Low.

Secretary, Mrs. W. L. Campbell.

At the Pittsburgh meeting W. D. McMaster was elected vice-chairman following the resignation of F. G. Low.

The committee was organized into five subcommittees as follows: Subcommittee I on Definitions and Nomenclature; Subcommittee II on Vapor Degreasing; Subcommittee III on Cold Cleaning; Subcommittee IV on Test Methods; and Subcommittee V on Industrial Hygiene.

This report has been submitted to letter ballot of the committee, which consists of 93 members; 19 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. D. McMaster,
Vice-Chairman.

Mrs. W. L. Campbell,
Secretary.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

REPORT OF COMMITTEE D-27
ON
ELECTRICAL INSULATING LIQUIDS AND GASES*

Committee D-27 on Electrical Insulating Liquids and Gases held one meeting this year, in Washington, D. C. on February 18, 19, and 20, 1959, at which time the committee was organized. At the last Annual Meeting, its predecessor, Subcommittee IV on Liquid Insulation of Committee D-9 on Electrical Insulating Materials, held a meeting in Boston, Mass., on June 23, 24, and 25, 1958, and a second meeting was held in Philadelphia, Pa., on October 29, 30, and 31, 1958. These last two meetings have been reported in the report of Committee D-9.

At the February 1959 meeting, Committee D-27 took the necessary steps to ratify its organization. A charter and governing by-laws were prepared under the jurisdiction of the temporary officers and Advisory Committee appointed by the Board of Directors of ASTM. A nominating committee was appointed by the temporary chairman, F. M. Clark, and the following permanent officers were elected for one-year terms to expire at the close of the Annual Meeting in 1960:

Chairman, F. M. Clark.
Vice-Chairman, E. R. Thomas.
Recording Secretary, C. A. Johnson.
Membership Secretary, R. M. Frey.

With installation of the elected officers, the chairman appointed subcommittee chairmen and, following their acceptance, held a meeting of the Advisory Committee in New York on March 25, 1959, at which the organization was approved as outlined under "Activities of Subcommittees." The subcommittee chairmen were empowered to organize the necessary sections to carry on the operations of the subcommittees, and to appoint section chairmen so that the work under way would not be delayed. It was agreed to transfer the membership of Subcommittee IV to Committee D-27, and to assign all section members of Subcommittee IV to the corresponding subcommittees and sections of Committee D-27.

Appointment of Liaison Representatives.—The chairman reappointed the following liaison representatives from Committee D-27:

J. G. Miller will report to Methods Subcommittees O and P as their representative on Committee D-2, Research Division X on Corrosion.

V. R. Mulhall will report to the Advisory Subcommittee as its representative on Committee E-11, Subcommittee I.

F. S. Oliver will report to Materials Subcommittees A and C on the activities of Subcommittee 5 on Dielectric Measurements, AIEE Committee on Special Instruments and Auxiliary Apparatus.

E. L. Raab will report to Materials Subcommittee A on the activities

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

TABLE I.—RESULTS IN DUPLICATE OF COOPERATIVE TESTS USING METHOD FOR SEDIMENT AND SOLUBLE SLUDGE IN INSULATING OILS.

Laboratory	Total Sediment, per cent		Organic Sediment, per cent		Inorganic Sediment, per cent		Soluble Sludge, per cent	
	Oil No. 1	Oil No. 2	Oil No. 1	Oil No. 2	Oil No. 1	Oil No. 2	Oil No. 1	Oil No. 2
A	0.05	0.18	0.05	0.15	0.01	0.03	0.07	0.04
	0.06	0.18	0.05	0.15	0.01	0.03	0.07	0.04
B	0.07	0.13	0.07	0.10	0.01	0.02	0.06	0.04
	0.06	0.11	0.06	0.10	0.01	0.02	0.04	0.04
C	0.05	0.17	..	0.14	..	0.03	0.09	0.06
	0.03	0.16	0.05	0.13	0.01	0.03	0.09	0.06
D	0.06	0.18	0.05	0.16	0.01	0.03	0.09	0.04
	0.06	0.18	0.06	0.16	0.01	0.02	0.08	0.04
E	0.04	0.14	0.04	0.13	0.01	0.01	0.06	0.03
	0.04	0.14	0.04	0.13	0.01	0.01	0.06	0.03

NOTE.—Two other laboratories cooperated in these tests but their data are not being reported because one laboratory submitted incomplete data, omitting results for organic and inorganic sediment; and the other laboratory used fritted glass crucibles instead of asbestos padded Gooch crucibles.

of Working Group 34, Subcommittee on Insulating Fluids, Committee on Transformers, AIEE.

R. G. Call will report to Methods Subcommittee O on the activities of the Power Station Chemistry Group, EEI.

J. C. Parker will report to Committee D-9, and Committee D-27 on the activities of the Subcommittee on Insulations, Insulated Conductor Committee, AIEE.

The committee will sponsor a joint symposium on November 18, 1959 at the Hotel Governor Clinton in New York City, with the membership of the Subcommittee on Insulations, Insulated Conductor Committee of the American Institute of Electrical Engineers. Papers on cable oils, cable insulating paper, and one expressing the opinions of the electrical utilities on the subject of cable insulation will be given. F. M. Clark will also speak on the activities of Committee D-27 and its predecessor, Subcommittee IV of Committee D-9, on the subject of cable oils.

Transfer of Jurisdiction of ASTM Methods.—ASTM methods previously under the jurisdiction of Subcommittee IV of Committee D-9 will now come under the custodianship of Committee D-27. This transfer will be effected by ASTM Headquarters for the following methods:

D 117 – 58, Methods of Testing Electrical Insulating Oils,

D 831 – 48, Test for Gas Content of Insulating Oils,

D 877 – 49, Test for Dielectric Strength of Insulating Oil of Petroleum Origin,

D 878 – 49, Test for Inorganic Chlorides and Sulfates in Insulating Oils,

D 901 – 56, Methods of Testing Askarels,

D 923 – 56, Method for Sampling Electrical Insulating Oils,

D 924 – 58, Test for Power Factor and Dielectric Constant of Electrical Insulating Oils of Petroleum Origin,

D 989 – 51, Test for Detection of Free Sulfur in Electrical Insulating Oils,

D 1040 – 58 T, Recommended Practice for the Purchase of Uninhibited Mineral Oil for Use in Transformers and in Oil Circuit Breakers,

D 1169 – 58, Test for Insulation Resistivity of Electrical Insulating Oils of Petroleum Origin,

D 1275 – 58, Test for Corrosive Sulfur in Electrical Insulating Oils,

D 1313 – 54, Test for Sludge Formation in Mineral Transformer Oil by High Pressure Oxidation Bomb,

D 1314 – 54 T, Test for Sludge Formation in Mineral Transformer Oil by Sludge Accumulation,

D 1315 – 54 T, Test for Water in Insulating Oils by Extraction,

D 1473 – 58 T, Test for 2,6-Ditertiary Butyl Paracresol in New Electrical Insulating Oils,

D 1524 – 58 T, Test by Visual Examination of Used Electrical Oils in the Field,

D 1533 – 58 T, Test for Water in Insulating Liquids (Karl Fischer Method),

D 1534 – 58 T, Test for Approximate Acidity of Used Electrical Insulating Oil of Petroleum Origin, and

D 1563 – 58 T, Test for Peroxide Number of Mineral Insulating Oils.

One method was under the joint jurisdiction of Committee D-2 and the former Subcommittee IV and will now be jointly controlled by Committees D-2 and D-27:

D 94 – 56 T, Test for Saponification Number of Petroleum Products by Color-Indicator Titration.

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Sediment and Soluble Sludge in Used Insulating Oils as appended hereto.[1] The method is a detailed procedure for separating and weighing soluble and insoluble sludge in a used oil. Cooperative test data using this method are shown in Table I.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method for Sampling Electrical Insulating Oils (**D 923 – 56**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society. The proposed revisions will make the method applicable to the sampling of cable oils from high- and low-pressure cables, splices, and terminals.

Section 7.—At the end of the section add the following:

For sampling under conditions of high humidity, and for sampling low- or high-pressure oil-filled cables under some conditions, special methods and containers may be advantageous. In such cases, if a special container is recommended in the selected method, it should be used.

Section 8(b).—Insert between the second and third sentences the following:

They should be kept sealed until immediately before sampling, and should be sealed again as soon as the sample is taken, to prevent contamination by dirt or moisture. In order to avoid confusion, samples taken should be properly identified as to location, apparatus, and so on, using appropriate company designations. To prevent breakage, the sample container after filling should be handled with care during transportation and storage.

Section 9(b).—Add the following to the end of the section:

To prevent condensation, a container which is colder than the ambient temperature should be heated until its temperature is above the ambient temperature. When samples must be taken under adverse humidity conditions, there is less chance of contamination if the sampling is done with the apparatus and method described in Section 3(*b*) of Methods of Test for Gas Content of Insulating Oils (ASTM Designation: D 831),[3] or one of the apparatuses and methods described in Appendix I, II, III, or IV of this specification. In the case of the more viscous oils that may be used in high-pressure cable and solid-type cable accessories when they are in containers, it may be necessary to heat the oils before samples can be taken. In such cases, the oil shall be heated with steam coils or equivalent heat sources where surface temperatures do not exceed 100 C.

---

[1] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 9.

[2] 1958 Book of ASTM Standards, Part 9.

[3] The new Appendices are included in the revised method which appears in the 1959 Supplement to Book of ASTM Standards, Part 9.

Section 13.—Add new Paragraphs (*e*) and (*f*) to read as follows:

(*e*) In some cases, special technique may be required for sampling low- or high-pressure oil-filled cables. In such cases, the procedure described in Section 3(*b*) of Methods of Test for Gas Content of Insulating Oils (ASTM Designation: D 831),[3] or one of the procedures described in Appendix I, II, III, or IV of this specification may be suitable.

(*f*) In the case of cables, it is suggested that sampling of oil under pressure be done in such a manner as to avoid the possibility of misleading test results which may be due to agitation of the oil.

New Appendices. — To the entire method, add the following Appendices I, II, III, and IV as appended hereto.[3]

WITHDRAWAL OF TENTATIVE REVISIONS OF STANDARD

With the approval of the revisions set forth above, the committee recommends the withdrawal of the tentative revisions issued Feb. 28, 1958 of Sections 7, 9(*b*), and 13 of the Standard Method for Sampling Electrical Insulating Oils (**D 923 – 56**).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Materials Subcommittee A on Mineral Oils (E. L. Raab, chairman) has been organized with five sections devoted to survey, transformer oils, cable oils, capacitor oils, and switch oils. Work is being brought to completion on limiting specifications for light and heavy cable oils. Transformer oils practices have been further strengthened by additions to Recommended Practice D 1040. Capacitor oils will follow the limits outlined for cable oils. The section on switch oils will determine test methods necessary for evaluation.

Materials Subcommittee B on Synthetic Fluids (T. K. Sloat, chairman) will continue its work on the development of tests for askarels. The sections of former Subcommittee IV of Committee D-9 will be transferred to the sections of this subcommittee, and new sections will be authorized as expanding work requires. A revision of Method D 901 was recently approved to include a test procedure for determining water content.

Materials Subcommittee C on Insulating Gases (R. B. Blodgett, chairman) is a new organization authorized for the study of insulating gases and to devise test procedures which may be required in the standardization of such materials. Sections have been established dealing with fixed gases, such as nitrogen, helium, hydrogen, etc., and on fluorides, as well as on combinations of fluorine and carbon. This new activity for the committee will broaden the field of dielectrics, and it is hoped that new members will be drawn from chemical companies engaged in this rapidly expanding operation. Many new methods will be required for the standardizing of procedures for testing these materials, and it will be necessary to draw on the experience of the new members for the technical requirements of these test methods.

Methods Subcommittee N on Electrical Tests (H. O. Bennett, chairman) has taken over the several sections of the former Subcommittee IV of Committee D-9, and will continue to develop procedures for dielectric strength, resistivity, stability of cable oil, and paper combinations, as well as methods for determining gas evolution.

Methods Subcommittee O on Chemical Tests (H. W. McCulloch, Jr., chairman) is continuing the work on oxidation and sludge tests for mineral insulating oils, corrosion tests, neutralization number, and the detection of metals. Revisions of a test method for determining additive content (D 1473) are being considered to make it applicable for determining the remaining additive in used insulating oils. Moisture tests are under revision, and are being adapted for use on askarels.

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Methods Subcommittee P on Physical Tests (J. C. Parker, chairman) is considering methods for refractive index, specific optical dispersion, gas content, coefficient of expansion, interfacial tension, sampling, and color. A method for measuring soluble and insoluble sludge has been approved this past year.

This report has been submitted to letter ballot of the committee, which consists of 100 members; 56 members returned their ballots, of whom 51 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. M. CLARK,
Chairman.

C. A. JOHNSON,
Recording Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-27 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Scavenger Content of Askarels (D 1701 – 59 T), and
Visual Examination of Used Askarels in the Field (D 1702 – 59 T).

Revision of Tentative Method of Test for:

Water in Insulating Oils by Extraction (D 1315 – 54 T).

These recommendations were accepted by the Standards Committee on September 15, 1959, and the new and revised tentative methods appear in the 1959 Supplement to Book of ASTM Standards, Part 9.

REPORT OF ALCA-ASTM JOINT COMMITTEE ON LEATHER*

The ALCA-ASTM Committee on Leather (joint with the American Leather Chemists' Assn.) held two meetings during the year: on November 13 and 14, 1958, in Philadelphia, Pa., and on March 19 and 20, 1959, in Washington, D. C.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Method of Determining the Bond Strength of Leather Belting as appended hereto.[1]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

Area of Leather (**D 1515 – 57 T**), and

Width of Leather (**D 1516 – 57 T**).

Tentative Definitions of:

Terms Relating to Leather (**D 1517 – 57 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature, Definitions, and Applicability (R. M. Lollar, chairman) is reviewing test methods in regard to definitions and applicability. Particular attention is being given to statements of significance and precision of all methods passing through the committee.

Subcommittee II on Sampling and Conditioning for Physical Testing (C. W. Mann, chairman) is preparing methods for sampling cattlehide side leather and small skins. A sampling procedure for interlaboratory tests will also be prepared.

Subcommittee III on Physical Testing (G. A. Butz, chairman):

Section 2 on Physical Dimensions (J. Naghski, chairman) is preparing a tentative method for thickness of units.

Section 3 on Tensile Properties (R. L. Young, chairman) is working on methods for tensile strength, stitch tear, breaking strength, ball and Mullen burst strength, and elongation. Particular emphasis is being placed on the significance statements and the precision and accuracy of the test methods.

Section 4 on Measurement of Water Resistance (R. G. Ashcraft, chairman) has drafted a method for static water absorption. A method for dynamic water penetration resistance of upper leathers has been prepared but needs further data on precision. Work is under way on methods for spray testing of garment leather, dynamic water absorption of light leathers, and penetration resistance of glove leathers. A method to determine water resistance of sole leather is now being evaluated by the industry, and a walking machine will be evaluated for the testing of Army combat boots containing rubber soles and manufactured by the vulcanizing process.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 6.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Section 5 on Surface Characteristics (R. Stubbings, chairman) is conducting an exposure test for light-fastness of leather which is now nearly completed. A method for cold crock resistance of upper leather is completed except for editorial changes. Other test methods being studied are methods for cold flex testing and one for the flex testing of finishes. Methods to evaluate crocking and scuffing are under way as are methods to determine stain resistance of leather.

Section 7 on Mechanical Leathers (A. N. Compton, chairman) prepared the Tentative Method of Determining the Bond Strength of Leather Belting referred to earlier, and is working on a method to determine area shrinkage of leathers when immersed in various fluids at elevated temperatures.

Section 8 on Deterioration (C. F. Dudley, chairman) is working on test methods to evaluate the perspiration resistance of white and colored leathers, also heat and dry cleaning stabilities of leather.

Section 10 on Miscellaneous Physical Properties (A. N. Kay, chairman) is working on methods for piping (break), grain cracking, stiffness, compressibility, wear resistance, creep (spread of soles) and run of glove leather.

Subcommittee IV on Research (M. M. Baldwin, chairman) is investigating methods related to comfort of leather: namely, water vapor adsorption from both rate and equilibrium standpoints, heat conductivity, cyclic stress-strain tensile properties and resiliency, electrical conductivity and heat of wetting. The committee is considering all types of nondestructive tests for testing leather as a continuing function. It has also been assigned the responsibility of providing suitable subjects among those under study by the committee for formal discussion during the meetings of the Joint Committee.

Subcommittee V on Editorial (T. Thorstensen, chairman) has concerned itself principally with the form and organization for presentation of test methods.

This report has been submitted to letter ballot of the committee, which consists of 57 members; 42 members returned their ballots, of whom 38 have noted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JOSEPH R. KANAGY,
Chairman.

C. W. MANN,
Secretary.

REPORT OF COMMITTEE E-1
ON
METHODS OF TESTING*

Committee E-1 on Methods of Testing held one meeting during the year: at Philadelphia, Pa., on March 31, 1959. Sixteen subcommittees and task groups met during the 1958 Annual Meeting of the Society in Boston, Mass. Subcommittee 28 on Microscopy met in Chicago, Ill., on June 11, 1958. Meetings were held of sixteen subcommittees and task groups during ASTM Committee Week in Pittsburgh, Pa., from February 2 to 6, 1959. Meetings of the following three subcommittees were held in New York, N. Y., on February 17 and 18, 1959: Subcommittee 17 on Thermometers, Subcommittee 18 on Hydrometers, and Subcommittee 21 on Metalware Laboratory Apparatus.

A new Subcommittee 32 on Thermocouples for Temperature Measurement was organized at a meeting in Philadelphia, Pa., on November 24, 1958, with J. W. Freeman, chairman and D. I. Finch, secretary. Four task groups have been organized under this subcommittee as follows: No. 1 on Standardization of Standard Thermocouple Materials, No. 2 on Newer Thermocouple Materials, No. 3 on Review of ASTM Methods, and No. 4 on Monograph on Use of Thermocouples.

Committee E-1 is sponsoring a Symposium on Microscopy[1] during the 1959 Annual Meeting of the Society. This Symposium comprises 10 papers and will be held in three sessions on June 25 and 26. Mr. F. F. Morehead is chairman of the Symposium Committee.

The committee records with sorrow the passing of one of its past chairmen, W. H. Fulweiler, on December 20, 1958. Mr. Fulweiler had a record of long and distinguished service to the Society and particularly to Committee E-1, having served as its chairman for 20 years from 1928 to 1947. He will be missed by his many friends in the committee who held him in deep affection and high esteem.

The committee also records with sorrow the passing of C. A. Neusbaum on August 28, 1958.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee E-1 presented to the Society, through the Administrative Committee on Standards, the following recommendations:

Revision of Tentative Methods of:

Test for Brinell Hardness of Metallic Materials (E 10 – 54 T), and

Test for Softening Point by Ring and Ball Apparatus (E 28 – 51 T).

Revision of Tentative Definitions:

Terms Relating to Methods of Mechanical Testing (E 6 – 58 T).

Revision of Tentative Specifications for:

ASTM Hydrometers (E 100 – 57 T).

Revision of Standard and Reversion to Tentative:

Specifications for Sieves for Testing Purposes (E 11 – 39), and

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Issued as separate publication *ASTM STP No. 257.*

Definitions of Terms Relating to Rheological Properties of Matter (E 24 - 42).

The revision of Tentative Method E 10 - 54 was accepted by the Standards Committee on September 9, 1958, and appears in the 1958 Book of ASTM Standards, Part 3. The revision of Tentatives E 6 - 58 T, E 24 - 42, and E 11 - 39 were accepted on September 9, 1958, and appear in the 1958 Book of ASTM Standards, Parts 4, 5, 7, 9, and 10. The revision of Tentative Method E 28 - 51 T was accepted on October 20, 1958, and appears in Parts 7, 8, and 9 of the 1958 Book of ASTM Standards. The revision of Tentative Specifications E 100 - 57 T was accepted on October 20, 1958 and appears in the 1958 Book of ASTM Standards, Parts 7 and 8.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications, method of test, and recommended practice as appended hereto:

Specifications for:

Gravity-Convection and Forced-Ventilation Laboratory Ovens,[2]

Method for Determination of:

Shear Modulus at Room Temperature,[3] and

Recommended Practice for:

Safe Use of Oxygen Combustion Bombs.[4]

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Method for Determination of Young's Modulus at Room Temperature (E 111 - 58 T):[5]

Section 2.—In Note 1 add the following at the end of the last sentence: "or, for certain materials, the determination may be made after a limited amount of prestressing and release of load. In these cases the conditions under which a significant value of Young's modulus can be determined should be prescribed in the product specification."

Specifications for ASTM Hydrometers (E 100 - 58 T):[6]

Table I.—Add the following requirements for a new hydrometer 101H for testing liquefied petroleum gas as requested by Committee D-2 on Petroleum Products and Lubricants:

ASTM Hydrometer No.	101H
Nominal Specific Gravity Range	0.500 to 0.650
Hydrometer Scale:	
Standard Temperature	60/60 F
Subdivisions	0.001
Intermediate Lines at	0.005
Main (numbered) Lines at	0.010
Scale Error at any Point not to Exceed	0.001
Scale Length	125 to 145 mm
Thermometer Scale:	
Range	30 to 90 F
Immersion	Total[a]
Subdivisions	1 F
Intermediate Lines at	5 F
Main (numbered) Lines at	10 F
Scale Error at any Point not to Exceed	0.5 F
Scale Length	50 to 70 mm
Total Length	354 to 366 mm
Body Diameter	19 to 22 mm
Stem Diameter	10.5 mm, min

[a] Thermometer bulb shall be embedded in a suitable heat-transfer medium enclosed within the body of the hydrometer.

Revise *Hydrometers 21H to 40H* by changing the requirement for body diameter from "12 to 18 mm" to "12 to 15 mm."

---

[2] The new tentative, bearing the designation E 145 - 59 T, appears in the 1959 Supplement to Book of ASTM Standards, Parts 6, 8, 9, and 10.

[3] The new tentative, bearing the designation E 143 - 59 T, appears in the 1959 Supplement to Book of ASTM Standards, Parts 3, 6, and 9.

[4] The new tentative, bearing the designation E 144 - 59 T, appears in the 1959 Supplement to Book of ASTM Standards, Parts 7, 8, and 9.

[5] 1958 Book of ASTM Standards, Parts 3, 4, 5, 6, and 9.

[6] 1958 Book of ASTM Standards, Parts 7 and 8.

Revise *Hydrometers 51H to 60H*, inclusive, to show that these are available for temperature ranges of 60 to 220 F, 30 to 180 F, and 0 to 150 F.

Revise *Hydrometers 71H to 74H* by changing the requirement for body diameter from "18 to 25 mm" to "23 to 27 mm."

Table II.—Add the following under the heading "Hydrometers for Light Liquids Other than Alcohol:"

Density and Specific Gravity	API Gravity, deg	Liquid
0.5073 (sp gr 60/60 F)	147.4	Pure grade propane[a]
0.5844 (sp gr 60/60 F)	110.6	Pure grade *n*-butane[a]

[a] The listed specific gravities are nominal values. Suitable materials of known specific gravity are available from Phillips Petroleum Co., Special Products Division, Bartlesville, Okla. Orders should carry the notation "For ASTM Hydrometer Standard—show specific gravity on label." The specific gravity value given on the label will be based on a spectrographic analysis of the material and will be accurate to ±0.0002. In the use of these materials care should be exercised to observe the safety precaution given in the Method of Sampling Liquefied Petroleum Gases (ASTM Designation: D 1265).

Section 11.—Add the following new Paragraph (*a*), relettering the present paragraphs accordingly: "11. (*a*) The thermometer shall be of the mercury-in-glass type, unless otherwise specified."

Add a new Paragraph (*f*) to read as follows:

> (*f*) The thermometer shall be calibrated in accordance with the procedures outlined in the Method of Testing and Standardization of Etched-Stem Liquid-in-Glass (ASTM Designation: E 77).

Method for Inspection and Verification of Hydrometers (E 126 – 57 T):[7]

Table I.—Make the same addition to this table as recommended above for Table II of Specifications E 100.

Section 7(a).—Change to read: "(*a*) Use either of the comparators described in Section A1 and A2 of the Appendix.

New Section.—Add as Section 9 a new procedure for verification of hydrometers as appended hereto.[8]

Appendix.—Add the following new section:

> A3. *Comparators*, suitable for checking hydrometers in liquids of high vapor pressure are shown in Fig. 2. The working pressure of the unit shall be not less than 200 psig.

New Figure.—Add the accompanying Fig. 1 as a new Fig. 2.[9]

TENTATIVE REVISION OF STANDARD

The committee recommends tentative specifications for three new thermometers to be published as a tentative revision of the Standard Specifications for ASTM Thermometers (E 1 – 58) as shown in the accompanying Table I. The weathering test thermometer was requested by Committee D-2 for use in testing liquefied petroleum gases. The two solidification point thermometers were prepared for Committee D-11 on Rubber and Rubber-like Materials for use in the Method of Test for Melting Range of Rubber Chemicals (D 1519 – 58 T).

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as indicated of the Standard Specifications for ASTM Thermometers (E 1 – 58)[10] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society:

---

[7] 1958 Book of ASTM Standards, Parts 4 and 7.

[8] The new procedure appears in the revised method, see 1959 Supplement to Book of ASTM Standards, Parts 4 and 7.

[9] The new figure is not included in this report but appears in the revised method, see 1959 Supplement to Book of ASTM Standards, Parts 4 and 7.

[10] 1958 Book of ASTM Standards, Parts 4, 7, and 8.

Section 4(b).—Change to read as follows by the addition of the italicized words:

> (*b*) When the top finish of the thermometer is specified in Table I as "glass ring," the outside diameter of the glass ring shall be not more than the *specified maximum* diameter of the stem and the inside diameter not less than 2 mm.

Section 6(a).—Replace the present requirements which reads "The width of graduation lines shall be not more than one fifth the graduation interval" by the following 4 groups. The first is an exception for thermometers 56C and 56F. In the case of the other three a maximum line width of 0.12 mm is recommended for thermometers which may be read to fractions of a division, many times with magnifying aids; 0.18 mm for thermometers which may be read to the nearest half division or where the congestion of the scale dictates the use of a line of moderate fineness; and finally 0.23 mm maximum for thermometers with more open scales usually read to the nearest division, oftentimes under adverse conditions, where a bold graduation is therefore desired.

Group 1. Maximum line width 0.10 mm: Thermometers 56C, 56F.

Group 2. Maximum line width 0.12 mm: Thermometers 14C, 14F, 26C, 28F, 29F, 30F, 44F, 45F, 46F, 47F, 48F, 50F, 51F, 52C, 53C, 62C, 62F, 63C, 63F, 64C, 64F, 65C, 65F, 66C, 66F, 67C, 67F, 68C, 68F, 69C, 69F, 70C, 70F, 72F, 73F, 74F, 89C, 90C, 91C, 92C, 93C, 94C, 95C, and 96C.

Group 3. Maximum line width 0.18 mm: Thermometers 1C, 1F, 2C, 2F, 3C, 3F, 12C, 12F, 15C, 15F, 16C, 16F, 17C, 17F, 18C, 18F, 19C, 19F, 20C, 20F, 21C, 21F, 22C, 22F, 23C, 24C, 25C, 33C, 33F, 34C, 34F, 35C, 35F, 36C, 43F, 49C, 54F, 61C, 61F, and 71F.

Group 4. Maximum line width 0.23 mm: Thermometers 5C, 5F, 6C, 6F, 7C, 7F, 8C, 8F, 9C, 9F, 10C, 10F, 11C, 11F, 13C, 27C, 37C, 38C, 39C, 40C, 41C, 42C, 57C, 57F, 58F, 59F, 60F, 75F, 76F, 77F, 78F, 79F, 80F, 81F, 82F, 83F, 84F, 85F, 86F, 87F, 88C, 88F, 97F, and 98F.

Table I.—Flash Point Thermometers 10C and 10F, 11C and 11F.—Change the scale errors to read as follows:

2.5 F up to 500 F 3.5 F over 500 F	1 C up to 260 C 2 C over 260 C

Petrolatum Melting Point Thermometers 61C and 61F.—Add a footnote to these thermometers to require the bulb bottoms to be essentially hemispherical.

New Method of Dimensioning Thermometers.—Revise the dimensional requirements of the thermometers in Table I to read as shown in the accompanying Table II. These changes will result in a new method of dimensioning thermometers which consists in locating both the bottom and top ranges from the bottom of the bulb. The advantages of this new method of dimensioning are as follows:

1. All ASTM thermometers will now meet NBS certification requirements for capillary clearances at top of range.
2. Manufacture will be facilitated.
3. The possibilities of international standardization will be increased.

EDITORIAL CHANGES

The committee recommends editorial changes in two tentatives as follows:

Definitions of Terms Relating to Methods of Mechanical Testing (E 6 - 58a T):[11]

Section 13.—Delete the note.

Method for Determination of Poisson's Ratio at Room Temperature (E 132 - 58 T):[5]

Section 4(b).—Add the following new Note 3 and new Footnote 6 after the first paragraph, renumbering subsequent Notes and footnotes accordingly:

---

[11] 1958 Book of ASTM Standards, Parts 3, 4, 5, 6, 9, and 10.

TABLE I.—PROPOSED SPECIFICATIONS FOR ASTM THERMOMETERS.

Name	ASTM Weathering Test[a]	ASTM Solidification Point	ASTM Solidification Point
ASTM Thermometer No.	99F	100C	101C
Immersion	35 mm[b]	76 mm	76 mm
Temperature Range	−55 to +40 F	145 to 205 C	195 to 305 C
Subdivisions	0.5 F	0.2 C	0.5 C
Longer Graduation Lines at Each	1 F	1 C	1 C
Graduations Numbered at Each Multiple of	5 F	2 C	5 C
Scale Error at any point when standardized shall not exceed	0.4 F	0.4 C	1.0 C
Standardization	−55, −30, 0, and +32 F in armor	145, 165, 185, 205 C	200, 250, 300 C
With Average Temperature of Emergent Mercury Column of	−10 F	(to be supplied by NBS)	(to be supplied by NBS)
Total Length	299 to 305 mm	365 to 375 mm	365 to 375 mm
Stem Diameter	6.5 to 8.0 mm[c]	6.0 to 7.0 mm	6.0 to 7.0 mm
Bulb Diameter	not less than 6.0 mm, not greater than stem	not less than 5.0 mm, not greater than stem	not less than 5.0 mm, not greater than stem
Bulb Length	15 to 20 mm	18 to 28 mm	18 to 28 mm
Contraction Chamber: Top of chamber to bottom of bulb, max	...	41 mm	41 mm
Bottom of Bulb to Graduation Line at Distance	−55 F 50 to 75 mm	140 C 116 to 130 mm	190 C 116 to 130 mm
Bottom of Bulb to Graduation Line at Distance	+40 F 244 to 265 mm	200 C 315 to 335 mm	300 C 315 to 335 mm
Expansion Chamber shall permit heating to	Elongated Type 150 F	250 C	330 C
Top Finish	[d]	Glass ring	Glass ring
Special Inscription on Thermometer	ASTM WEATHERING 35 MM IMM	ASTM SOLID PT 76 MM IMM	ASTM SOLID PT 76 MM IMM

---

[a] A suitable mercury-thallium alloy shall be used as the actuating liquid.

[b] The immersion line shall be visible in the case opening after assembly. The immersion shall be measured from the bottom of the bulb rather than from the bottom of the armor.

[c] The stem shall be either the round or lens-front type.

[d] The top finish shall permit assembly in a 12-in. spark-resisting metal armor with open face and the distance from bottom of the bulb to bottom of the armor shall be not more than 13 mm.

TABLE II.—PROPOSED CHANGES IN ASTM THERMOMETER SPECIFICATIONS.[a]

ASTM Thermometer No.	6C | 6F	7C | 7F	8C | 8F	9C | 9F
Total Length	227 to 237 mm	381 to 391 mm	381 to 391 mm	282 to 292 mm
Bottom of Bulb to Graduation Line at	20 C | 68 F	300 C | 572 F	400 C | 752 F	100 C | 212 F
Distance	182 to 196 mm	333 to 354 mm	333 to 354 mm	221 to 237 mm
ASTM Thermometer No.	10C | 10 F	11C | 11F	12 C | 12 F	13C
Total Length	282 to 292 mm	303 to 313 mm	415 to 425 mm	164 to 174 mm
Bottom of Bulb to Graduation Line at	360 C | 680 F	360 C | 680 F	100 C | 212 F	170 C
Distance	227 to 245 mm	237 to 254 mm	354 to 379 mm	120 to 134 mm
ASTM Thermometer No.	14C | 14F	15C | 15F	16C | 16F	17C | 17F
Total Length	372 to 382 mm	392 to 402 mm	392 to 402 mm	270 to 280 mm
Bottom of Bulb to Graduation Line at	80 C | 176 F	80 C | 176 F	200 C | 392 F	26.7 C | 80 F
Distance	315 to 335 mm	333 to 354 mm	333 to 354 mm	217 to 236 mm
ASTM Thermometer No.	18C	18F	19C | 19F	20C | 20F
Total Length	270 to 280 mm	270 to 280 mm	270 to 280 mm	270 to 280 mm
Bottom of Bulb to Graduation Line at	42 C	108 F	56.7 C | 134 F	64.4 C | 148 F
Distance	215 to 234 mm	217 to 236 mm	217 to 236 mm	217 to 236 mm
ASTM Thermometer No.	21C | 21F	22C	22F	23C
Total Length	270 to 280 mm	270 to 280 mm	270 to 280 mm	207 to 217 mm
Bottom of Bulb to Graduation Line at	86.7 C | 188 F	103 C	218 F	28 C
Distance	217 to 236 mm	214 to 233 mm	217 to 236 mm	160 to 177 mm
ASTM Thermometer No.	24C	25C	26C	27C
Total Length	232 to 242 mm	207 to 217 mm	458 to 468 mm	296 to 306 mm
Bottom of Bulb to Graduation Line at	54 C	105 C	140 C	182 C
Distance	185 to 202 mm	160 to 177 mm	376 to 398 mm	246 to 266 mm
ASTM Thermometer No.	28F	29F	30F	33C | 33F
Total Length	300 to 310 mm	300 to 310 mm	300 to 310 mm	414 to 424 mm
Bottom of Bulb to Graduation Line at	102.5 F	132.5 F	212.5 F	40 C | 104 F
Distance	193 to 218 mm	193 to 218 mm	193 to 218 mm	352 to 377 mm
ASTM Thermometer No.	34C | 34F	35C | 35F	36C	37C
Total Length	414 to 424 mm	414 to 424 mm	400 to 410 mm	390 to 400 mm
Bottom of Bulb to Graduation Line at	105 C | 221 F	170 C | 338 F	68 C	52 C
Distance	358 to 379 mm	358 to 384 mm	350 to 370 mm	335 to 360 mm
ASTM Thermometer No.	38C	39C	40C	41C
Total Length	390 to 400 mm	390 to 400 mm	390 to 400 mm	390 to 400 mm
Bottom of Bulb to Graduation Line at	78 C	102 C	126 C	152 C
Distance	334 to 359 mm	335 to 360 mm	335 to 360 mm	335 to 360 mm
ASTM Thermometer No.	42C	43F	44F	45F
Total Length	390 to 400 mm	413 to 428 mm	300 to 310 mm	300 to 310 mm
Bottom of Bulb to Graduation Line at	255 C	−29 F	71.5 F	79.5 F
Distance	335 to 360 mm	224 to 286 mm	193 to 218 mm	193 to 218 mm

---

[a] Changes in Thermometers 5C and 5F appear at end of Table II.

TABLE II.—(*Concluded*).

ASTM Thermometer No.	46F	47F	48F	49C
Total Length	300 to 310 mm	300 to 310 mm	300 to 310 mm	300 to 310 mm
Bottom of Bulb to Graduation Line at	124.5 F	142.5 F	182.5 F	70 C
Distance	193 to 218 mm	193 to 218 mm	193 to 218 mm	245 to 270 mm
ASTM Thermometer No.	50F	51F	52C	53C
Total Length	463 to 473 mm	463 to 473 mm	157 to 167 mm	184 to 194 mm
Bottom of Bulb to Graduation Line at	101 F	116 F	5 C	10 C
Distance	412 to 438 mm	412 to 438 mm	106 to 128 mm	130 to 150 mm
ASTM Thermometer No.	54F	56C | 56F	57C | 57F	58F
Total Length	307 to 317 mm	580 to 590 mm	282 to 292 mm	300 to 305 mm
Bottom of Bulb to Graduation Line at	212 F	35 C | 95 F	50 C | 122 F	120 F
Distance	245 to 275 mm	510 to 550 mm	233 to 252 mm	255 to 275 mm
ASTM Thermometer No.	59F	60F	61C | 61F	71F
Total Length	300 to 305 mm	300 to 305 mm	376 to 386 mm	350 to 360 mm
Bottom of Bulb to Graduation Line at	180 F	500 F	125 C | 257 F	70 F
Distance	255 to 275 mm	255 to 275 mm	312 to 342 mm	290 to 310 mm
ASTM Thermometer No.	72F	73F	74F	75F
Total Length	300 to 310 mm	300 to 310 mm	300 to 310 mm	403 to 413 mm
Bottom of Bulb to Graduation Line at	2.5 F	−37.5 F	−62.5 F	32 F
Distance	142 to 167 mm	142 to 167 mm	142 to 167 mm	346 to 362 mm
ASTM Thermometer No.	76F	77F	78F	79F
Total Length	403 to 413 mm	270 to 280 mm	270 to 280 mm	270 to 280 mm
Bottom of Bulb to Graduation Line at	2 F	265 F	315 F	365 F
Distance	346 to 362 mm	217 to 236 mm	217 to 236 mm	217 to 236 mm
ASTM Thermometer No.	80F	81F	82F	83F
Total Length	270 to 280 mm	270 to 280 mm	159 to 165 mm	168 to 174 mm
Bottom of Bulb to Graduation Line at	415 F	465 F	220 F	160 F
Distance	217 to 236 mm	217 to 236 mm	135 to 143 mm	144 to 152 mm
ASTM Thermometer No.	84F	85F	86F	87F
Total Length	378 to 387 mm	305 to 314 mm	164 to 170 mm	169 to 175 mm
Bottom of Bulb to Graduation Line at	175 F	300 F	350 F	400 F
Distance	354 to 362 mm	275 to 289 mm	134 to 148 mm	139 to 153 mm
ASTM Thermometer No.	88C | 88F	89C	90C	91C
Total Length	282 to 292 mm	365 to 375 mm	365 to 375 mm	365 to 375 mm
Bottom of Bulb to Graduation Line at	200 C | 400 F	10 C	30 C	50 C
Distance	233 to 252 mm	315 to 335 mm	315 to 335 mm	315 to 335 mm
ASTM Thermometer No.	92C	93C	94C	95C
Total Length	365 to 375 mm	365 to 375 mm	365 to 375 mm	365 to 375 mm
Bottom of Bulb to Graduation Line at	70 C	90 C	110 C	130 C
Distance	315 to 335 mm	315 to 335 mm	315 to 335 mm	315 to 335 mm
ASTM Thermometer No.	96C	97F	98F	5C | 5F
Total Length	365 to 375 mm	300 to 305 mm	300 to 305 mm	226 to 236 mm
Bottom of Bulb to Graduation Line at	150 C	120 F	180 F	49 C | 120 F
Distance	315 to 335 mm	255 to 275 mm	255 to 275 mm	195 to 205 mm

NOTE 3.—If exceptions are provided in the product specification so that extensometers of types other than those covered in Method E 83 are used, it may be necessary to apply corrections, for example, the correction for the transverse sensitivity[6] of bonded-wire resistance gages.

---

[6] C. C. Perry and H. R. Lissner, "The Strain Gage Primer," McGraw-Hill Book Co., Inc., New York, N. Y., pp. 141 to 146 (1955).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[12]

ACTIVITIES OF SUBCOMMITTEES AND TASK GROUPS

Subcommittee 1 on Calibration of Mechanical Testing Machines and Apparatus (B. L. Wilson, chairman).—A new Task Group on Calibration of Testing Machines Under 2000 lb Capacity (D. C. Scott, Jr., chairman) has been organized at the request of Committees D-11 on Rubber and Rubber-Like Materials and D-13 on Textile Materials.

Subcommittee 2 on Effect of Speed in Mechanical Testing (W. Ramberg, chairman).—Mr. Ramberg found it necessary to resign as chairman following his appointment as Scientific Attaché at the U. S. Embassy in Rome. He has been succeeded by L. K. Irwin, the present secretary. Mr. D. E. Driscoll was appointed as the new secretary of this subcommittee.

Consideration is being given to problems associated with high-speed tests using existing testing machines. It is expected that a task group will be organized to undertake this project.

The Task Group on Non-metallic Materials under the chairmanship of F. G. Tatnall is organizing 7 subgroups, each under the direction of a specialist, to prepare for further studies on the effect of speed in mechanical testing of: (1) plastics and polymers, (2) rubber, textiles and fibers, (3) ceramics and cermets, (4) cement and concrete, (5) paper, (6) wood and sandwich construction, and (7) asphalt. General rules to guide the subgroups will be formulated. There will be a study of the historical background of specifications that have been formulated to meet testing requirements where effect of speed of testing is a factor. The task group will confine itself to investigation of the effect of speed of testing of non-metallic materials at room temperature only.

Subcommittee 3 on Elastic Properties and Definitions on Mechanical Testing (G. R. Gohn, chairman).—The Task Group on Elastic Constants (W. Ramberg, chairman) developed the new Method of Test for Shear Modulus at Room Temperature appended to this report.[3] The next project of this task group is a method for determining Young's modulus at elevated temperature. Mr. Ramberg resigned from the chairmanship of this task group and has been replaced by James A. Miller, National Bureau of Standards.

A task group under H. L. Fry drafted definitions relating to creep and relaxation testing which were included in the Tentative Definitions of Terms Relating to Methods of Mechanical Testing (E 6 – 58a T). Revisions of the terms for fatigue and ductility are under review. An attempt is being made to reconcile differences in meaning in terms used in the plastics and metals fields. Liaison has been established with the American Society for Metals to avoid conflict in definitions.

Subcommittee 5 on Compression Testing (J. A. Miller, chairman).—The additions of sections on the determination of yield strength, yield point, and compressive strength are under consideration for inclusion in the Tentative Methods of

---

[12] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Compression Testing of Metallic Materials (E 9 – 52 T).[13]

Four new task groups are being organized on (1) review of methods and specifications, (2) bibliography, (3) compression testing of brittle materials, and (4) compression testing of plastic materials.

Subcommittee 6 on Indentation Hardness (R. H. Heyer, chairman).—The Task Group on Rockwell Hardness - Tensile Strength Conversions for Steel (Joint ASTM and SAE Committee) completed an extensive series of tests in which nine laboratories participated. Recommendations for changing certain conversion tables are being prepared for consideration by the subcommittee.

A hardness conversion between Knoop Hardness and Diamond Pyramid Hardness has been developed for nickel and high-nickel alloys.

The Task Group on Rockwell Testing has developed a "round work correction table" for Rockwell testing up to 1-in. diameter using the $\frac{1}{16}$-in. ball indenter for inclusion in the Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 57 T).[13]

The consolidation of the Rockwell and Superficial Rockwell sections of Methods E 18 into one section is under study.

Subcommittee 7 on Impact Testing (W. W. Werring, chairman).—Consideration is being given to subsize impact test specimens for possible inclusion in the Tentative Methods for Notched-Bar Impact Testing of Metallic Materials (E 23 – 56 T).

Subcommittee 9 on Rheological Properties (W. F. Fair, Jr., chairman).—A new task group on Brookfield Viscosity (S. E. Berger, chairman) has been organized at the request of Committee D-20 on Plastics. The organization meeting will be held in June during the Annual Meeting.

Subcommittee 10 on Sieves (L. V. Judson, chairman).—An important revision of the Standard Specifications for Sieves for Testing Purposes (E 11) was completed subsequent to the 1958 Annual Meeting. The revised specifications were reverted to tentative and have recently been approved as American Standard Z23.1—1959 by the American Standards Assn. The revision, which incorporated more uniform requirements for nominal wire diameters used in the manufacture of U. S. sieves, resulted from considerations given to the sieve specifications by the Committee on Sieves (ISO/TC 24). It changed the former nominal permissible range for wire diameters for each sieve to a single recommended nominal wire diameter with a permissible deviation expressed in percentage for range of sieve opening.

Subcommittee 11 on Sub-Sieve Testing (L. T. Work, chairman).—Following the Symposium on Particle Size Measurement held at the 1958 Annual Meeting it was decided to organize the following four task groups on: Sampling and Dispersion (R. H. Berg, chairman), Choice of Test Methods (Katherine Mather, chairman), Data and Interpretation (J. H. Calbeck, chairman), and Sedimentation Methods (K. T. Whitby, chairman).

Subcommittee 12 on Methods for Density (C. T. Collett, chairman).—A proposed method of test for bulk density of abrasive grain[13] has been developed in cooperation with the Abrasive Grain Assn.

Consideration is being given to the organization of two task groups—one on methods for density of liquids and the other on methods for density of solids, powders, and granular materials.

A task group is also being organized to undertake a review and revision of the Standard Definition of Terms Relating to Specific Gravity (E 12 – 27).

---

[13] See Editorial Note, p. 568.

Subcommittee 14 on Conditioning and Weathering (F. M. Gavan, chairman).—The Standard Specifications for Enclosures and Servicing Units for Tests Above and Below Room Temperature (D 1197 – 56) are being transferred by Committees D-9 on Electrical Insulating Materials and D-20 on Plastics to the jurisdiction of this subcommittee. The specifications will be reviewed and after revision will be issued as an "E" standard.

Work is continuing on a revision of the Method of Determining Relative Humidity (D 337 – 34) which will also be issued as an "E" standard, and also on revision of the Methods of Test for Measuring Water Vapor Transmission of Materials in Sheet Form (E 96 – 53 T).

Other subjects under consideration are: (1) plastic chip method for weatherometer, (2) fadeometer tests, and (3) outdoor exposure tests.

Subcommittee 17 on Thermometers (R. M. Wilhelm, chairman).—After many years of faithful service, Mr. R. Mason Wilhelm has found it necessary for reasons of personal health to resign as chairman of the subcommittee. Mr. Robert D. Thompson was elected to replace Mr. Wilhelm as chairman.

Specifications for several new thermometers have been developed in response to working committee requests. Weathering Test Thermometer 99F is to be used by Committee D-2 for tests on LPG materials. It is expected that this specification will also be adopted by the Natural Gasoline Association of America. At the request of Committee D-11 on Rubber and Rubber-Like Products, specifications for Solidification Point Thermometers 100C and 101C were prepared. These thermometers will be used for determining the melting range of rubber chemicals.

The new method of dimensioning ASTM thermometers introduced last year has been approved by Committees D-1, D-2, D-3, D-5, D-12, D-15, D-16, and D-23 for thermometers of interest to them. Adoption of revisions in these specifications completes this significant change to improve the quality of the thermometers and simplify manufacturing problems.

Revisions have been made in specifications for graduation line width to bring them more in accord with use requirements. Specifications for thermometer rings have been established to eliminate confusion in interpretation of requirements. Relaxation of accuracy tolerances of certain high-temperature flash point thermometers has been made in line with NBS certification practice and requirements of the Institute of Petroleum (London).

Cooperative work with ISO/TC 48 on Laboratory Glassware and Related Apparatus has continued in an endeavor to develop revised specifications for precision thermometers suitable for adoption as an international standard.

Projects under study include: a revision of standardization temperatures; the development of a new space-saving format for thermometer specifications; a comparative study of proposed ISO and present ASTM bulb stability tests; and the development of a new series of short-range, high-temperature distillation thermometers.

Subcommittee 18 on Hydrometers (R. D. Thompson, chairman).—Specifications and methods of test of a special form of thermohydrometer for specific gravity measurements of LPG products were developed at the request of Committee D-2. It is understood that these specifications will also be adopted by the Natural Gasoline Association of America.

The revision of specifications of plain form hydrometers resulting from standardization activities in ISO/TC 48 reported last year was confirmed by Committees D-2 and D-15 and was subsequently approved by the Society.

Revisions in the temperature ranges of API thermohydrometers have been made to reconcile long-standing differences between ASTM and API requirements. Certain dimensional changes were also made to improve readability and facilitate manufacture.

The accuracy tolerances of API short-form hydrometers, 21H to 40H, were relaxed in keeping with use requirements. At the same time the body diameters were revised to eliminate certain unuseable sizes.

A number of revisions of Specifications for ASTM Hydrometers (E 100) were made to clarify several confusing features.

Under study is a request from Committee D-2 to develop specifications for short-range, high-sensitivity instruments to be used in testing LPG products.

Subcommittee 19 on Glassware Laboratory Apparatus (J. J. Moran, chairman).—During the past year, task groups of the subcommittee have assisted several committees of the Society in the preparation of specifications for glass apparatus, as follows: apparatus for dilution test of crankcase oils, in a Revision of Method D 322; a buret for dilute tetraethyllead fluid, for Committee D-2; and additional distillation receivers for determination of water by distillation, to be added to Specifications E 123 – 56 T.

The subcommittee is investigating the desirability of publishing standard specifications for common items of volumetric glassware, and methods of calibrating volumetric glassware.

Subcommittee 21 on Metalware Laboratory Apparatus (E. L. Ruh, chairman) prepared the Specifications for Gravity-Convection and Forced-Ventilation Laboratory Ovens and the Recommended Practice for Safe Use of Oxygen Combustion Bombs appended to this report.

A new task group on Safety Requirements for Pressure Cylinders for Elastomer Aging (G. C. Maassen, chairman) has been organized.

Subcommittee 25 on Shear and Torsion Tests (R. W. Fenn, Jr., chairman) reviewed over sixty replies to a questionnaire on shear and torsion tests from other technical committees of the Society. After study of this information it was decided to sponsor a Symposium on Shear Testing to be held at the 1960 Annual Meeting. The papers to be presented at this symposium should be helpful to the subcommittee in its efforts to write recommended shear tests procedures and also provide a useful reference on the many aspects of shear testing. The objectives of this Symposium are: (1) to obtain detailed descriptions of new or existing shear-test methods as used on a wide variety of materials; (2) to determine the test variables in each shear-test method and to evaluate the effect of the variables on the shear-test results; (3) to describe the advantages and disadvantages of the various shear-test techniques, and (4) to compare shear-test results obtained using different test techniques or conditions. The Symposium Committee consists of R. W. Fenn, Jr. (chairman), The Dow Chemical Co.; A. J. Herzog, of Wright Air Development Center; and R. B. Clapper, of Lockheed Aircraft Corp.

Subcommittee 27 on Low-Temperature Testing of Elastomers and Plastics (F. M. Gavan, chairman).—The Task Group on Correlation of Test Methods (C. K. Chatten, chairman) is preparing a final paper which will present a survey and correlation of various low-temperature tests of elastomers and plastics, the details of which were reported in four papers published in the ASTM BULLETIN.

The Task Group on Rheological Creep (L. Boor, chairman) has completed a report on "Study of Rheological Testing of Elastomers at Low Temperatures, Part II," which will be submitted for publication in the ASTM BULLETIN.

A paper covering a survey of hardness tests of elastomers at low temperatures is in preparation by another task group under the chairmanship of Dan Pratt.

Subcommittee 28 on Microscopy (F. G. Foster, chairman).—The Symposium on Microscopy[1] to be held at this year's Annual Meeting is sponsored by this subcommittee.

Further study is being made of the proposed specifications and methods of test for glass covers and slides used in microscopy and the specifications for immersion fluids for use in microscopy.

Work is continuing on a bibliography of ASTM methods on microscopy and on a list of definitions of terms used in microscopy.

Subcommittee 29 on Microchemical Apparatus (A. Steyermark, chairman) is in the process of completing specifications to be recommended as standards for the apparatus used in connection with determination of nitrogen by both the micro-Kjeldahl and micro-Dumas methods.[13] The apparatus to be used for the microdetermination of carbon and hydrogen is next on the agenda of the subcommittee.

This report has been submitted to letter ballot of the committee, which consists of 32 voting members; 24 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. WEBBER,
Chairman.

P. J. SMITH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Apparatus for Microdetermination of Nitrogen by Kjeldahl Method (E 147 - 59 T), and
Apparatus for the Microdetermination of Nitrogen by the Dumas Method (E 148 - 59 T).

New Tentative Method of Test for:

Bulk Density of Abrasive Grains (E 153 - 59 T).

Revision of Tentative Methods of:

Compression Testing of Metallic Materials (E 9 - 52 T), and
Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 - 57 T).

These recommendations were accepted by the Standards Committee on October 27, 1959, with the exception of the new Tentative Specifications E 147 which were accepted on September 15, 1959. The new and revised tentatives appear in the 1959 Supplement to the Book of ASTM Standards.

REPORT OF COMMITTEE E-2
ON
EMISSION SPECTROSCOPY*

Committee E-2 on Emission Spectroscopy held two meetings during the year: in Boston, Mass., on June 25, 1958, and in Pittsburgh, Pa., on March 5, 1959. The Executive Subcommittee met at these times as well as in Ottawa, Canada, on September 15, 1958.

A half-day Symposium on Excitation[1] will be presented on June 24, 1959, at Atlantic City, N. J., during the Annual Meeting of the Society.

A three-day Symposium on Unsolved Problems in Spectroscopy[2] is being planned in cooperation with Committee E-13 on Absorption Spectroscopy, to be held at the Third Pacific Area National Meeting, San Francisco, Calif., October 12 through 16, 1959.

The Regulations for ASTM Committee E-2 on Emission Spectroscopy and Recommendations for Preparation of Spectrochemical Methods has been accepted and published by Committee E-2.

The committee plans a third edition of the Methods for Emission Spectrochemical Analysis, to be published in 1960.

The committee consists of 200 members. This represents no change since the last Annual Meeting. A membership campaign is planned in order to get more members from the area west of the Mississippi River and from Canada.

SUGGESTED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the following suggested methods as appended hereto:[3]

Suggested Method for Spectrochemical Analysis of:

Antimony by the Point-to-Plane Spark Technique (SM 6-16), and

Magnesium-Base Alloys by the Point-to-Plane Spark Technique Using a Grating Spectrograph (SM 7-13).

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Spectrochemical Analysis of Zinc-Base Alloys and High-Grade Zinc by the Solution - D-C Arc Technique (E 27 - 53 T):[4]

Section 3.—Revise Paragraphs (*e*) and (*f*) to read as follows:

(*e*) ***Developing Equipment.***—Developing, fixing, washing, and drying equipment shall conform to the requirements of the Recommended Practices for Photographic Processing in Spectrochemical Analysis (ASTM Designation: E 115).

(*f*) ***Electrodes.***—The electrodes shall be prepared from high-purity graphite rods found by spectrochemical test to be free of elements to be determined in the sample. The upper electrode shall be type C-6 as listed in Recommended Practices for Designation of Shapes and Sizes of Preformed Graphite Electrodes (ASTM Designation: E 130). The lower electrode shall be type S-6 in Recommended Practices E 130. Just prior to use, the electrodes shall be preburned in the d-c arc for a minimum of 30 sec.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Issued as separate publication *ASTM STP No. 259.*

[2] To be issued as separate publication *ASTM STP No. 269.*

[3] See pp. 573 and 577.

[4] 1956 Book of ASTM Methods for Chemical Analysis of Metals.

Section 8(b).—Revise to read as follows:

> (*b*) *Emulsion Calibration*.—Calibrate the emulsion in accordance with the Recommended Practices for Photographic Photometry in Spectrochemical Analysis (ASTM Designation: E 116).

Section 9.—Revise to read: "9. Process the emulsion in accordance with Recommended Practices E 115."

Tentative Method for Spectrochemical Analysis of Tin Alloys for Minor Constituents and Impurities (E 51 – 43 T):[4]

Section 3.—Revise Paragraph (*f*) to read as follows:

> (*f*) *Electrodes*.—The electrodes shall be of highest purity graphite. The lower positive electrode shall be of type S-8 in Recommended Practices for Designation of Shapes and Sizes of Preformed Graphite Electrodes (ASTM Designation: E 130). The electrode shall be waterproofed with a solution of halowax in CCl_4. The upper electrode shall be type C-6 in Recommended Practices E 130.

Add a new Paragraph (*g*) to read as follows:

> (*g*) *Developing Equipment*.—Developing, fixing, washing, and drying equipment shall conform to the requirements of the Recommended Practices for Photographic Processing in Spectrochemical Analysis (ASTM Designation: E 115).

Section 6.—Revise the first sentence to read "Fill the cups of the lower electrodes described in Paragraph 3(*f*) flush to the brim with portions of samples and standards."

New Section.—Insert a new Section 7 to read as follows, renumbering the present Section 7 as Section 8:

> 7. *Photographic Processing*.—Process the emulsion in accordance with the Recommended Practices E 115.

Tentative Method for Spectrochemical Analysis of Pig Lead by the Point-to-Plane Spark Technique (E 117 – 56 T):[4]

Section 8.—Revise the last two sentences to read: "Insert a C-2 type electrode as listed in Recommended Practices for Designation of Shapes and Sizes of Preformed Graphite Electrodes (ASTM Designation: E 130) in the spark stand and adjust the analytical gap to 3 mm. Make the lower electrode electrically positive."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Recommended Practices for:

Photographic Processing in Spectrochemical Analysis (**E 115 – 56 T**),

Photographic Photometry in Spectrochemical Analysis (**E 116 – 56 T**), and

Designation of Shapes and Sizes of Preformed Graphite Electrodes (**E 130 – 57 T**).

Tentative Methods for Spectrochemical Analysis of:

Nickel Alloys by the Powder-D-C Arc Technique (**E 129 – 57 T**), and

Aluminum and Aluminum-Base Alloys by the Point-to-Plane Spark Technique (**E 101 – 53 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus and Equipment (A. J. Mitteldorf, chairman) is preparing tentative practices on describing and specifying the excitation source and a report on the specifications for the spectrograph, compiling a summary of the characteristics of the various emulsions used throughout the world, testing cut film of SA No. 1 emulsion, and studying the possibility of describing spectrographs and microphotometers in terms of properties rather than manufacturer's names.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Fundamental Practices (R. W. Smith, chairman) is preparing a suggested practice for the use of statistics in spectrochemical analysis and a revision of the two tentatives on photographic processing and photometry, developing a recommended basic practice in flame photometry, assembling a bibliography in flame photometry, preparing a recommended basic practice in X-ray spectroscopy, assembling a bibliography in X-ray spectroscopy, planning a cooperative test for X-ray analysis of steel, and compiling a list of unsolved problems in spectrochemical analysis. A report on the cooperative test on the X-ray fluorescence of powdered samples has been finished.[6]

Subcommittee III, Editorial (E. B. Owens, chairman) has reviewed eleven manuscripts for publication, attempted to establish 50 new definitions of spectrographic terms, reviewed suggested changes to the Regulations for ASTM Committee E-2 on Emission Spectroscopy and Recommendations for Preparation of Spectrochemical Methods, and assisted in the publication of the Annual Report.

Subcommittee IV on Electrodes, Pure Materials, Reagents, and Standards (W. J. Edgar, chairman) is maintaining a current list of all standards and pure materials available throughout the world and is preparing a revision of the Tentative Recommended Practice for Designation of Shapes and Sizes of Preformed Graphite Electrodes (E 130 – 57 T).

Subcommittee V on Copper, Nickel, and Their Alloys (A. W. Young, chairman) is assisting the National Bureau of Standards in preparing copper-alloy standard samples, revising E-2 SM 5-2 (Spectrochemical Analysis of Wrought Copper Alloys by the D-C Arc Technique) with a view toward establishing it as a tentative, preparing suggested methods pertaining to the analysis of nickel-base alloy, and processing three new suggested methods.

Subcommittee VI on Lead, Tin, Zinc, and Related Materials (J. F. Murphy, chairman) has approved the Suggested Method for Spectrochemical Analysis of Antimony Metal by the Point-to-Plane Technique (E-2 SM 6-16), and the suggested revisions in Methods E 27, E 51, and E 117 referred to earlier in the report. The subcommittee is planning to test cooperatively E-2 SM 6-1, E-2 SM 6-4, and E-2 SM 6-9, is investigating the possibilities of using solution methods for the analysis of cadmium and bismuth metals, and is also writing direct reader methods for zinc and for lead. This subcommittee also cooperatively tested SM 6-15 (Spectrochemical Analysis of Zinc Die-Casting Metal and High-Purity Zinc by the Cast Pin and Point-to-Plane Technique), and has compiled a report on the results of this test.[7]

Subcommittee VII on Aluminum, Magnesium, and Their Alloys (F. R. Potter, chairman) is cooperatively testing E-2 SM 7-10 (Suggested Method for Spectrochemical Analysis of Aluminum and Its Alloys by the Point-to-Plane Spark and Intermittent Arc Techniques, Using a Recording Photoelectric Spectrometer) and a new suggested method for the spectrochemical analysis of magnesium alloys for calcium by the flame photometer technique. They have set up a new task group to gather and develop X-ray fluorescence methods. The Suggested Method for Spectrochemical Analysis of Magnesium-Base Alloys by the Point-to-Plane Spark Technique Using a Grating Spectrograph (E-2 SM 7-13) referred to earlier has been approved by the subcommittee.

---

[6] This report has been published in *Applied Spectroscopy*, Vol. 13, No. 1, p. 3 (1959); reprints are available on request at ASTM Headquarters.

[7] Copies of this report are available on request at ASTM Headquarters.

Subcommittee VIII on Titanium, Zirconium, and Related Alloys (N. E. Gordon, chairman) is considering two new suggested methods for the analysis of titanium and four new suggested methods for the analysis of zirconium, and is working cooperatively with the Materials Branch of the Atomic Energy Commission and the National Bureau of Standards to obtain zirconium and zirconium alloy standards.

Subcommittee IX on Ferrous Alloys (J. F. Woodruff, chairman) is cooperatively testing the following three methods:

1. Proposed Tentative Method for the Spectrochemical Analysis of Plain Carbon and Low-Alloy Steels by the Point-to-Plane Technique,
2. Proposed Tentative Method for the Spectrochemical Analysis of Plain Carbon and Low-Alloy Steel by the Rod-to-Rod Technique, and
3. Proposed Tentative Method for Spectrochemical Analysis of Stainless-Type Steels by the Point-to-Plane Spark-Initiated Intermittent Arc Technique Using a Recording Photoelectric Spectrometer.

Seven suggested methods are being reviewed.

Subcommittee X on Glass, Ceramics, Alkalies, and Cementitious Materials (W. M. Hazel, chairman) is considering six new suggested methods for the analysis of nonmetallic materials, alumina, cement, and glass.

Subcommittee XI on Slags, Ores, and Other Miscellaneous Nonmetallic Materials (S. R. Wiley, chairman) is reviewing the following four suggested methods for the spectrochemical analysis of: (1) blast-furnace and steel making slags; (2) miscellaneous powder samples by the d-c arc technique; (3) cerium minerals for the rare-earth elements and thorium by the d-c arc technique; and (4) titanium oxide by the d-c arc technique.

This report has been submitted to letter ballot of the committee, which consists of 160 members; 88 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

DAVID L. FRY,
Chairman.

CYRUS FELDMAN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Recommended Practices for:

Photographic Processing in Spectrochemical Analysis (E 115 – 56 T), and
Photographic Photometry in Spectrochemical Analysis (E 116 – 56 T).

These recommendations were accepted by the Standards Committee on November 6, 1959, and the revised tentative recommended practices are available as separate reprints.

APPENDIX I

SUGGESTED METHOD FOR SPECTROCHEMICAL ANALYSIS OF ANTIMONY BY THE POINT-TO-PLANE SPARK TECHNIQUE[1]

SUBMITTED BY E. J. DUNN, JR.,[2] J. P. CESTARO,[2] AND W. VON WEISENSTEIN[2]

E-2 SM 6-16

Scope

1. (*a*) This method covers the determination of eight elements in antimony by a spark technique. It is used primarily to check the purity of virgin antimony. The elements and ranges of concentration covered by the method are as follows:

Element	Concentration Range, per cent
Lead	0.01 to 0.6
Bismuth	0.01 to 0.5
Arsenic	0.004 to 0.5
Tin	0.008 to 0.4
Copper	0.003 to 0.4
Iron	0.003 to 0.1
Nickel	0.001 to 0.08
Silver	0.002 to 0.01

(*b*) This method is designed for inspection testing of chill cast disks or miscellaneous antimony samples on which a flat surface area of 1 in. in diameter or greater can be prepared, and which are sufficiently massive to prevent overheating under the action of the spark.

Summary of Method

2. The sample is excited with a controlled spark, using the point-to-plane technique. The spectra are recorded photographically. Intensity ratios of selected pairs of analytical lines and antimony internal standard lines are determined photometrically, and elemental concentrations are read from analytical curves.

Apparatus

3. (*a*) *Sample Molds.*—Iron spectrographic molds designed to produce disks $2\frac{3}{4}$ in. in diameter and $\frac{1}{4}$ in. in thickness.

(*b*) *Petrey Stand* or other suitable means for holding the sample during excitation.

(*c*) *Excitation Source.*—A source unit capable of giving an overdamped discharge generated by a condensed arc. A synchronously rotating switch is provided in the discharge circuit.

(*d*) *Filter*, 6 per cent transmission, placed in a filter slide mounted close to the film.

(*e*) *Spectrograph.*—A 1.5-m grating spectrograph having sufficient resolving power and dispersion to separate clearly the analytical lines from other lines in the spectrum of the sample in the spectral region 2150 to 4350 A. The 1.5-m grating has a reciprocal linear dispersion, in the first order, of 7 A per mm. Prism spectrographs can also be used for these analyses.

(*f*) *Developing Equipment*, as described in the Recommended Practices for Photographic Processing in Spectrochemical Analysis (ASTM Designation: E 115).[3]

(*g*) *Microphotometer*, capable of sufficient stability to measure spectral lines with a precision of ±1.5 per cent for transmittance values between 10 and 90 per cent. It may be either of the recording or nonrecording type.

---

[1] This suggested method has no official status in the Society, but is published as information only. The method is based on the experience of the submitters. Comments are solicited.

[2] National Lead Co. Research Laboratories, Brooklyn 1, N. Y.

[3] Available as a separate reprint.

(*h*) *Calculating Equipment.*—A calculating board with emulsion calibration scales capable of converting microphotometer readings to log intensity ratios. Analytical scales may be employed to convert log intensity ratios to per cent concentration.

Reagents and Materials

4. (*a*) *Counter Electrode.*—Regular grade ¼-in. graphite rod tipped with a 120-deg cone.

TABLE I.—ANALYTICAL LINE PAIRS.

Element	Analytical Line, A	Internal Standard Line, A	Concentration Range, per cent	Concentration Index, per cent	Slope, deg
Lead	Pb 2823.189	Sb 2858.044	0.06 to 0.6	0.38	56
	Pb 2833.069	Sb 2858.044	0.02 to 0.1	0.042	55
Bismuth	Bi 2938.298	Sb 2858.044	0.01 to 0.5	0.14	56
Arsenic	As 2860.452	Sb 2858.044	0.004 to 0.5	0.31	57
Tin	Sn 3262.328[a]	Sb 2858.044	0.03 to 0.4	0.29	55
	Sn 2839.989	Sb 2858.044	0.008 to 0.08	0.015	54
Copper	Cu 3273.962[a]	Sb 2858.044	0.003 to 0.4	0.080	69
Iron	Fe 3047.605	Sb 2858.044	0.003 to 0.1	0.12[b]	64.5
Nickel	Ni 3050.819	Sb 2858.044	0.001 to 0.08	0.015	60
Silver	Ag 3280.683	Sb 2858.044	0.002 to 0.01	0.018[b]	54

[a] Six per cent transmittance filter.
[b] Extrapolation.

(*b*) *Flux.*[4]

(*c*) *Metals for Preparation of Standards.*—High-purity, metallic form of antimony and the elements listed in Table I.

(*d*) *Photographic Emulsion.*—Eastman Spectrum Analysis No. 1 or equivalent.

(*e*) *Photographic Processing Solutions,* as described in Recommended Practices E 115.

Preparation of Standards

5. (*a*) All standards shall be prepared in the following manner: Prepare a master alloy by adding twice the maximum concentration of each element listed in Section 1 (*a*) to molten antimony (allow for 10 per cent loss). Maintain the temperature at approximately 800 C in the graphite crucible, using a reducing gas-air flame. Plunge the addition elements beneath the surface of the bath with forceps and stir with a graphite rod until the solution is uniformly molten. Cast in an iron spectrographic mold. Prepare standards in a like manner by addition of appropriate amounts of the above concentration of alloy to molten antimony to cover the percentage test range desired. Cast standards in iron spectrographic molds and number in the order of casting. Machine the standards on a lathe to give a clean, smooth, flat surface.

(*b*) Analyze the prepared disk standards in accordance with the Methods for Chemical Analysis of Antimony Metal (ASTM Designation: E 86)[5] and evaluate for segregation by spectrochemical analysis.

Preparation of Samples

6. (*a*) Machine the sample carefully on a lathe to give a clean, smooth, flat surface.

(*b*) For irregularly-shaped samples,

---

[4] Dow 230 Magnesium Flux, or equivalent, has been found satisfactory for this purpose.

[5] 1956 Book of ASTM Methods for Chemical Analysis of Metals.

chipped or drilled samples, and odd pieces, combine the portions by heating in a small silica crucible under the cover of a flux. Melt the flux first; then introduce the pieces of metal, making sure that the flux covers the metal at all times. As soon as the sample is uniformly molten, allow to chill and solidify. The flux also solidifies and is easily separated from the disk. Heat approximately enough sample to form one disk. If the amount of sample is small, use a smaller diameter crucible. Antimony melts at 630.5 C; it is preferable to keep the sample temperature below 650 C during the period required to make it molten.

Electrode System

7. The upper electrode is the specimen described. Mount the upper electrode with the flat surface horizontal. Use the counter electrode C-2 as described in the Recommended Practice for Designation of Shapes and Sizes of Preformed Graphite Electrodes (ASTM Designation: E 130).[5a] Clasp the counter electrode vertically in opposition to the flat surface of the upper electrode. Make the lower electrode electrically positive and adjust the analytical gap to 3 mm.

Excitation and Exposure

8. Produce and record the spectra according to the following conditions:

(*a*) *Electrical Parameters.*—A condensed arc source should be used.[6] The parameters should be set as follows:

Voltage, d-c, v	940
Capacitance, μf	20
Inductance, μh	200
Resistance, ohm	10
Current, avg, d-c, amp	1.2
Discharges per sec	60
Sample polarity	negative

NOTE.—The values of voltage, capacitance, inductance, resistance, and current are those read on the meters and switches of the condensed arc source. Such factors as resistance of the leads, stray capacitance, and the inductance of the resistors are thus disregarded. Other settings are as follows:

Initiator power	high
Phase, charge versus discharge, deg	180
Discharge point control, deg	90
Initiator circuit	continuous

TABLE II.—DATA ON PRECISION OF METHOD.

Element	Average, Concentration, per cent	Coefficient of Variation[a]	Number of Determinations
Lead	0.10	2.6	10
Bismuth	0.10	1.9	10
Arsenic	0.13	1.1	10
Tin	0.080	3.3	10
Copper	0.10	3.5	10
Iron	0.004	6.5	10
Nickel	0.02	6.3	10
Silver	0.0073	4.0	10

[a] Coefficient of variation, ν, in this method, is calculated as follows:

$$\nu = \frac{100}{\bar{X}}\sqrt{\frac{\Sigma d^2}{n - 1}}$$

where:
$\bar{X}$ = average concentration, per cent
d = difference of the determination from the mean, and
n = number of determinations.

(*b*) *Exposure Conditions:*

Spectral region, A	2150 to 4350
Filter region, A	3245 to 3305
Filter transmittance, per cent	6.0
Slit width, mm	0.050
Slit length, in.	0.054
Grating gate[a]	6-6
Spark preburn period	none
Spark exposure period, sec	approximately 30

[a] Numerical values given apply to the Applied Research Laboratories 1.5-m spectrograph (10-10 indicates a full open grating). Optimum values for any other spectrograph may be different.

(*c*) *Exposure Index.*—Adjust the exposure time to maintain the transmittance of the antimony line at 2858.04 A at approximately 15 per cent.

[5a] Methods for Emission Spectrochemical Analysis (1957).

[6] Such a source may be purchased from Applied Research Laboratories, Glendale, Calif. It is sold under the trade name Multisource.

Photographic Processing

9. Process the emulsion in accordance with Section 13 (*b*) or (*d*) of the Recommended Practices for Photographic Processing in Spectrochemical Analysis (ASTM Designation: E 115).[3]

Photometry

10. Read the transmittances for the analytical lines, the internal standard lines, and the emulsion calibration lines on a projection microphotometer. Select line pairs appropriate to the mode of excitation and the concentration range from the list in Table I.

Calibration

11. (*a*) *Emulsion Calibration.*—Calibrate the emulsion by one of the methods described in the Recommended Practices for Photographic Photometry in Spectrochemical Analysis (ASTM Designation: E 116).[3]

(*b*) *Preparation of Analytical Curves.*—Prepare analytical curves for each element in the standards by plotting the logarithm of concentration as ordinate as a function of the logarithm of intensity ratio as abscissa. Use a minimum of twelve determinations of each element in each standard for obtaining the average intensity ratio. Place eight analytical curves prepared from this data for the various elements on one sheet of heavy paper according to the scales on the calculating board.

(*c*) *Analytical Curve Correction.*—Place two standards, one of a high concentration and one of a low concentration, on each film. Use the intensity ratio values from the two standards to position the draft paper on the calculating board. This automatically takes care of any small drift in the analytical curves.

Calculations

12. Convert the transmittances of the analytical lines and internal standard lines to log intensity ratios by means of the film calibration curve prepared in accordance with Section 11 (*a*). Convert the log intensity ratios to concentration by reference to the appropriate analytical curve prepared in accordance with Section 11 (*b*). This is accomplished by placing the sheet of paper having the eight analytical curves for antimony on the calculating board. Set its position by the intensity values of the two standards on the film. Read the concentration of the element according to the instructions of the particular type of calculating board.[7] With this calculating board, once the curves and scales are properly set on the board, the concentration is read directly from the transmission value of each element. Report the average of duplicate determinations.

Precision and Accuracy

13. (*a*) Table II indicates the precision of the method obtained using a standard as the unknown.

(*b*) Comparisons of accuracy indicate that all spectrochemical determinations in the ranges given are equal or superior in accuracy to routine wet chemical determinations.

DISCUSSION

The electrical conditions chosen for this method yield an extremely stable spark which samples in a uniform $\frac{1}{4}$-in. diameter circle on the surface of the unknown.

The standard disks should be very carefully handled. Antimony is very brittle and will fracture easily if dropped.

---

[7] The Direct Reading Calculator has been found satisfactory for this purpose, see R. D. Dewey, "Direct Reading Calculator," *Journal of Applied Spectroscopy*, Vol. 7, No. 2, May, 1953, p. 87.

APPENDIX II

SUGGESTED METHOD FOR SPECTROCHEMICAL ANALYSIS OF MAGNESIUM-BASE ALLOYS BY THE POINT-TO-PLANE SPARK TECHNIQUE USING A GRAPHING SPECTROGRAPH[1]

SUBMITTED BY J. F. MURPHY[2]

E-2 SM 7-13

Scope and Application

1. (*a*) This method provides for the determination of alloying constituents and some residual elements in magnesium-base alloys in the ranges of concentrations shown:

Element	Concentration Range, per cent
Aluminum	0.50 to 12.5
Thorium	0.10 to 8.0
Zinc	0.30 to 6.0
Zirconium	0.10 to 3.0
Manganese	0.008 to 0.60
Calcium	0.05 to 0.50
Silicon	0.01 to 0.50
Lead	0.005 to 0.10
Copper	0.002 to 0.10
Tin	0.001 to 0.10
Iron	0.005 to 0.02

(*b*) This method is designed for analysis of AZ and HK alloys. The sample must be large enough so that a surface area of ½-in. diameter or greater may be prepared for sparking and of sufficient mass to prevent excessive overheating in the spark. The method is limited in that it lacks sufficient sensitivity for the determination of various trace elements. Other methods, such as the Suggested Method For Spectrochemical Analysis of Magnesium-Base Alloys by the Point-to-Plane Spark Technique (E-2 SM 7-4)[3] and the Suggested Method For Spectrochemical Analysis of Magnesium and Its Alloys by the Pin-to-Pin Spark Technique (E-2 SM 7-11)[3] provide for the determination of these trace impurities.

Summary of Method

2. A controlled spark excitation of a smooth, flat surface by the point-to-plane technique is employed. A hemispherically-tipped graphite counter electrode is used in the lower electrode holder. The spectra are photographed on a calibrated emulsion. The intensity ratios of selected analytical lines and magnesium internal standard lines are determined photometrically, and the concentrations of the elements sought are read from analytical curves relating log intensity ratio to log concentration.

Apparatus

3. (*a*) *Sample Mold.*—A steel mold designed to produce a disk 2½ in. in diameter and ½ in. in thickness.

(*b*) *Excitation Source.*—A high-voltage, air-interrupted, condensed spark unit, or equivalent, having the source parameters described in Section 8(*a*).[4]

(*c*) *Spectrograph.*—A grating spectrograph having a reciprocal linear dispersion of approximately 5 A per mm or a spectrograph having sufficient resolving power and linear dispersion to separate clearly the analytical lines in the spectrum of the sample in the spectral region 2200 to 3400 A. The spectrograph shall be illuminated by focusing an image of the spark discharge on the grating or collimating lens.

---

[1] This suggested method has no official status in the Society, but is published as information only. The method is based on the experience of the submitter. Comments are solicited.

[2] ACF Industries, 336 Woodward Rd., Albuquerque, N. Mex.

[3] "Methods for Emission Spectrochemical Analysis," Am. Soc. Testing Mats. (1957).

[4] The parameters given in Section 8(*a*) are those of an excitation source manufactured by Jarrell-Ash Co., Newtonville, Mass.

(*d*) *Developing Equipment.*—A thermostatically-controlled, agitating developing machine, washer, and warm-air drier.

(*e*) *Microphotometer.*—Any microphotometer having acceptable precision and accuracy.

(*f*) *Calculating Equipment.*—A calculating board to convert microphotometer readings to logarithmic ratios.

Reagents and Materials

4. (*a*) *Electrodes.*—Lower electrodes of high-purity graphite rods similar to electrode C-5 in the Recommended Practice for Designation of Shapes and Sizes of Preformed Graphite Electrodes (ASTM Designation: E 130).[4a]

(*b*) *Photographic Emulsion.*—Eastman Spectrum Analysis No. 1 or equivalent.

(*c*) *Photographic Processing Solutions,* as described in the Recommended Practices for Photographic Processing in Spectrochemical Analysis (ASTM Designation: E 115).[4a]

Standards

5. *Standard Samples.* — Secondary standards in the form of chill-cast disks and extruded bars are available commercially.[5]

Preparation of Standards and Samples

6. (*a*) Prepare a smooth, flat surface on both the standards and the samples by turning on a lathe or by use of belt grinder. Both standards and samples should be prepared in the same manner for any given test.

(*b*) Prepare a cast disk from samples that are not of the right size or shape to facilitate preparation of a surface for sparking. Melt the sample and cast into the sample mold as described in Section 3(*a*).

Electrode System

7. Make the sample the upper electrode and the high-purity graphite rod the lower electrode. Use an analytical gap of 4 mm.

Excitation and Exposure

8. Produce and record the spectra according to the following conditions:

(*a*) *Electrical Parameters:*[4]

Input voltage (line), v.	220
Output voltage (Powerstat), v.	185
Input voltage (spark transformer), v.	85
Discharge voltage, v.	18 000
Capacitance, μf.	0.005
Inductance, μh.	310
Primary resistance, dial setting.	5
Secondary resistance, ohm.	residual
Radio-frequency current, amp.	4.5
Number of discharges per cycle.	8

(*b*) *Exposure Conditions:*

Spectral region, A.	2200 to 3400
Slit width, mm.	0.030
Slit height, mm	2
Spark preburn period, sec.	5
Spark exposure period, sec.	30
Filters.	as needed

(*c*) *Exposure Index.*—Maintain the transmittance of the Mg 3073.99 A line at approximately 20 per cent.

(*d*) *Replicate Exposures.*—Make duplicate exposures for each sample along with two or more control standards also run in duplicate.

Photographic Processing

9. Process the emulsion in accordance with the Recommended Practices for

---

[4a] Available as a separate reprint.

[5] Report on Standard Samples and Related Materials for Spectrochemical Analysis," Am. Soc. Testing Mats. (1955). (Issued as separate publication *ASTM STP No. 58-C.*)

Photographic Processing in Spectrochemical Analysis (ASTM Designation: E 115).[4a]

Photometry

10. Measure the transmittance of the analytical line pairs and the calibration lines with a projection microphotometer. Select line pairs in Table I appropriate to the concentration of the respective elements present.

Calibration

11. (*a*) *Emulsion Calibration.*—Calibrate the emulsion by one of the methods described in the Recommended Practices for Photographic Photometry in Spectrochemical Analysis (ASTM Designation: E 116).[4a]

(*b*) *Preparation of Analytical Curves.*—Convert the transmittance of the analytical lines and the internal standard

TABLE I.—ANALYTICAL LINE PAIRS.

Element	Analytical Line, A	Concentration Range, per cent	Concentration Index, per cent
Aluminum	Al I 2367.06	2.5 to 12.5	11.10
	Al 2567.99	0.50 to 10.0	5.15
	Al I 2660.39	0.50 to 5.0	3.85
Thorium	Th 3325.13	0.50 to 8.0	7.70
	Th 3180.20	0.10 to 6.0	5.10
Zinc	Zn I 3345.57	1.5 to 6.0	3.05
	Zn I 3282.33	0.55 to 5.0	2.62
	Zn 3302.94	0.70 to 3.0	2.47
	Zn II 2557.96	0.30 to 2.6	1.95
	Zn I 3302.59	0.35 to 2.6	1.40
	Zn I 3345.02	0.35 to 1.5	0.80
Zirconium	Zr II 3306.28	0.30 to 3.0	1.6
	Zr II 3279.26	0.10 to 1.2	1.08
Manganese	Mn 2610.20	0.08 to 0.60	0.45
	Mn 2705.74	0.08 to 0.60	0.50
	Mn II 2701.70	0.05 to 0.50	0.42
	Mn 2889.58	0.02 to 0.50	0.38
	Mn II 2949.20	0.008 to 0.10	0.064
Calcium	Ca II 3179.33	0.05 to 0.50	0.267
Silicon	Si I 2528.52	0.04 to 0.50	0.38
	Si I 2516.12	0.01 to 0.20	0.125
	Si I 2881.58	0.01 to 0.15	0.055
Lead	Pb I 2833.07[a]	0.005 to 0.10	...
Copper	Cu I 3273.96	0.005 to 0.10	0.047
	Cu I 3247.54	0.002 to 0.05	0.025
Tin	Sn I 2839.99[a]	0.001 to 0.10	...
Iron	Fe II 2599.40[a]	0.005 to 0.02	...
Magnesium	Mg 3073.99	internal standard	...

[a] Visual determinations are made for lead, tin, and iron.

lines to log intensity ratios, using the emulsion calibration curve. Prepare analytical curves by plotting the log intensity ratio against the log concentration for each of the respective elements. Prepare a separate curve for each of the elements in each of the alloys.

TABLE II.—DATA ON PRECISION OF METHOD.

Alloy	Element	Average Concentration, per cent	Coefficient of Variation[a]	Number of Determinations
AZ31	Aluminum....	3.15	2.56	50
HK31	Thorium......	3.26	2.48	50
AZ31	Zinc..........	1.12	4.03	50
HK31	Zirconium....	0.82	3.69	50

[a] Coefficient of variation, ν, in this method, is calculated as follows:

$$\nu = \frac{100}{\overline{X}}\sqrt{\frac{\Sigma d^2}{n-1}}$$

where:

$\overline{X}$ = average concentration, in per cent,

d = difference of the determination from the mean, and

n = number of determinations.

Calculations

12. Determine the log intensity ratio for each analytical line pair from the calibration curve. Obtain the percentage concentration by referring this figure to the previously prepared appropriate analytical curve. Report the average of duplicate determinations.

Precision and Accuracy

13. (a) *Precision.*—The data on the precision of the method as listed in Table II was obtained over a six-month period.

(b) *Accuracy.*—The accuracy of the method depends upon the reliability of the standards used. Results obtained by this method as compared to results obtained by standard wet chemical methods show an average deviation for each element tested of from 4 to 6 per cent of the amount present.

REPORT OF COMMITTEE E-3
ON
CHEMICAL ANALYSIS OF METALS*

Committee E-3 on Chemical Analysis of Metals held one meeting during the year: on June 25, 1958, in Boston, Mass., in conjunction with the Annual Meeting of the Society. Divisional and other tasks groups held individual sessions on June 22 to June 25, 1958.

It is the intention of the committee to publish a new 1960 edition of "Book of ASTM Methods for Chemical Analysis of Metals." This, as well as previous publications, is made possible by the continued interest of the membership. Additional work in nuclear areas is under way or being considered. Task forces on both columbium and zirconium are very active. Requests have been made for the committee to examine chemical and isotopic methods for the analysis of uranium.

To expand the useful function of the committee further, it has been resolved that the subject of methods for the chemical analysis of ores be included as a part of the activity of Committee E-3.

A review was made of the roster of the committee. As a result, many of the inactive members have resigned and have been replaced. Additional active members will be sought to cover the expanding obligations of Committee E-3.

NEW TENTATIVE

The committee recommends for publication as tentative the Methods for Chemical Analysis of Zirconium and Zirconium-Base Alloys as appended hereto.[1]

REVISION OF TENTATIVE

The committee recommends the revision as appended hereto[1] of the Tentative Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 – 57 T)[2] and continuation of the method as tentative. The revision consists of the addition of a new photometric method for the determination of phosphorus.[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Editorial Subcommittee (J. L. Hague, chairman).—Meetings of the subcommittee were held at Boston on June 23, 1958, and at ASTM Headquarters on November 25, 1958. Detailed editing of a proposed style manual for Committee E-3 was completed, and copies distributed to the committee membership with the yearly preprints. Division editorial groups were set up for each of the method-writing divisions of the committee, and proposed schedules established for the preparation of a new edition of the "Book of ASTM Methods for Chemical Analysis of Metals," to appear in 1960.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] The new and revised tentatives have been issued as separate reprints.

[2] Issued as a separate reprint.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Division S on Sampling (A. C. Holler, chairman).—Due to the need for sampling methods for the newer metals and alloys, the division was subdivided into three subcommittees. These are: (1) Ferrous Subcommittee, for the sampling of ferrous metals and alloys; (2) Non-Ferrous Subcommittee, for the sampling of non-ferrous metals and alloys; and (3) Miscellaneous Metals Subcommittee, for the sampling of the newer metals and alloys such as lithium, molybdenum, titanium, etc.

Ferrous Subcommittee (J. B. Armstrong, chairman).—A method for the sampling of tubular products is complete and ready for balloting. The subcommittee discussed the method for the sampling of ductile iron with reference to the samples taken for the carbon determination.

Methods of Sampling Ferro-Alloys (E 32) was subjected to editorial review. A task group was appointed to study the method for the surface cleaning and drilling billets as covered in Method E 59.

Some questions regarding the sampling of pig iron were discussed. The chairman will contact Committee A-3 on Cast Iron regarding this.

Non-Ferrous Subcommittee.—At present the subcommittee is not actively engaged in any project, although consideration needs to be given to the revision of Methods E 88 covering the sampling of cast non-ferrous metals to bring them in line with current practice.

Miscellaneous Metals Subcommittee (John Danaczko, chairman).—This newly formed subcommittee will be studying the sampling methods for titanium sponge (in cooperation with Subcommittee IX of Committee B-2) and the newer metals such as lithium, molybdenum, etc., in cooperation with Subcommittee VIII of Committee B-2.

Division M on Miscellaneous Metals (G. Porter, chairman).—A meeting of Division M was held at Boston, Mass., on June 24, 1958. Due to the resignation of Mr. R. M. Fowler as Chairman, Mr. Porter has been appointed to serve as chairman of the division until the June 1959 election.

The number of people actively serving on the task groups and the amount of work accomplished has far exceeded expectations. This has been especially true in the case of the high-purity refractory metals.

A nominating committee consisting of Messrs. Goward (chairman), Vigo, and Kall has been appointed for the division election to be held at the June Annual Meeting.

Columbium Metal

Methods for the determination of carbon, nitrogen, oxygen, hydrogen, tantalum, titanium, silicon, iron, boron, molybdenum, tungsten, and zirconium in columbium have been collected in draft form from the task force members and distributed to element subgroup chairmen for rewriting, reproduction, and distribution. Of these methods, those for nitrogen, tantalum, titanium, molybdenum, tungsten, boron, and iron have been rewritten and distributed by the respective subgroup chairmen for round-robin testing. Six columbium samples have been subjected to preliminary analysis and selected numbers of these samples have been distributed for round-robin analysis. The nitrogen, tantalum, and titanium subgroup members have these samples for analysis. The subgroup on iron is performing analyses to determine the selection of one or two methods for this element. The oxygen subgroup has prepared a rod form standard for oxygen analysis. This standard has been shown to be homogeneous and has been distributed for round-robin analysis.

Metal Powders

Experimental work has been completed on the methods for the chemical analysis of metal powders and the hydrogen loss of copper powder. These methods are now undergoing editorial revision. Further experimental work is being done in connection with the methods for hydrogen loss of iron powder and the determination of insoluble matter in copper and iron powders.

Oxygen in Molybdenum Metal

An organizational meeting of this task group was held at Pittsburgh, Pa., on March 2, 1959. This problem had previously received considerable attention and work under the auspices of the Office of Naval Research. The original workers volunteered to serve on this task force and some of the samples are still available. Upon completion of the work on molybdenum a similar project may be undertaken on tungsten metal.

Titanium

Samples of titanium alloys were prepared and distributed to 11 members of the Task Group on Aluminum in Titanium Alloys. Three gravimetric methods were also distributed for evaluation: a double-fluoride method, a phosphate method, and a sodium hydroxide-oxine method.

Zirconium

Experimental work on methods for hydrogen, aluminum, iron, chromium, nickel, copper, manganese, and nitrogen has been completed and the proposed tentative is appended hereto.[1] Work is nearing completion on tin, titanium, molybdenum, and carbon methods. Additional work is being done on procedures for oxygen, lead, chloride, phosphorus, magnesium, sodium, cadmium, and uranium. Meetings of the group were held in Washington, D.C., on December 1, 1958 and in Pittsburgh, Pa., on March 2, 1959.

Other task force activities include cooperating with Committee E-2 in validating spectrographic standards, and cooperating with the Bureau of Standards in establishing standards for the industry. The membership of the committee now includes all sponge producers, all core vendors, and most melters and fabricators actively engaged in the nuclear zirconium industry.

Division F on Ferrous Metals (J. J. Furey, chairman).—One meeting of Division F was held at Boston, Mass., on June 24, 1958. The plan inaugurated in 1957 of holding informal meetings of the various task forces was again carried out and proved very successful.

At the formal meeting the task force chairmen presented reports of the projects under their jurisdiction which are summarized briefly:

Low Carbon (less than 0.01 per cent) in Steel

A letter and questionnaire were submitted to all task force chairmen requesting detailed information on methods and equipment used by each laboratory. Methods used by General Electric Co. and Union Carbide Metals Co. have been distributed. It is expected that the first draft of a tentative procedure will be ready for committee consideration at the 1959 Annual Meeting.

Ferroboron

Work has been completed by the task force and a method is being drafted. The National Bureau of Standards is issuing a standard sample of ferroboron based on the results obtained by the task force.

Ferrocolumbium

Methods are being developed in conjunction with the National Bureau of Standards. A 100-lb sample of the alloy has been sent to the Bureau for homogeneity tests and for evaluation of the ion exchange procedure for columbium and tantalum. It is expected that samples and methods of analysis will be distributed to the task force soon.

Manganese

A pyrophosphate method for manganese in ferromanganese and silicomanganese is being evaluated as an alternate method for the bismuthate method currently appearing in the 1956 Book of ASTM Methods for Chemical Analysis of Metals. Samples of both alloys have been distributed but all results have not been received.

Cobalt in High-Cobalt Alloys

The results of a method which eliminated need for a soda separation have been received, and the spread is too great to warrant further consideration. Additional work is now under way on a method involving a soda-bromine separation followed by a ferricyanide titration. Ion exchange will also be investigated.

Hydrogen in Steel

Hydrogen-charged nickel samples were prepared and distributed to the task force. The results were not encouraging. The spread between the laboratories involved was as great or greater than that obtained on the original steel samples. A new approach is now being considered.

Aluminum in Stainless Steel (Photometric Method)

An effort will be made to adapt the Armco procedure, with some modification, to the determination of aluminum. A method developed by Inland Steel Co. will be tried also, as will a method submitted by R. S. Kelly for aluminum above 0.50 per cent.

Nitrogen in Steel

A method to cover soluble and insoluble nitrogen is the objective of the task force. A photometric method has been distributed to task force members together with four samples. Another sample of high-nitrogen stainless will be prepared and distributed.

Copper in Steel (Photometric Method)

The work has been completed and the method is being finally revised.

Low Sulfur in Steel (0.0005 to 0.001 per cent) by Combustion

Discussion by the task force indicated the general belief that fundamental work must first be done to establish information on (*a*) blanks contributed by boats or crucibles, (*b*) sulfur absorbed and held by boats or crucibles, and (*c*) nature of compounds responsible for loss of sulfur and the methods for recovery or conversion to a measurable form. Work is continuing along these lines.

Phosphorus in Steel (Photometric Method)

The task force has completed its work; the method has been approved for publication, as appended hereto.[1] The supporting cooperative data for the new method have been filed with ASTM Headquarters and are available on request.

Tribute was paid to Messrs. Flickinger and Pitzer, both active members of Division F, who passed away during the year.

A nominating committee consisting of Messrs. Armstrong, Melnick, Van Tyne, and Holler (chairman) was appointed for Division F. They will bring in a slate of officers at the next Annual Meeting.

Division N on Non-Ferrous Metals (R. G. Ernst, chairman).—During the past year, work has continued on the completion of several methods which have been under study for some time. These include the rhodamine-B method for antimony, the determination of zinc in aluminum-base alloys, the photometric determination of selenium and tellurium, the determination of cadmium and zinc in mixtures of the two, the determination of traces of aluminum in slab zinc, and the determination of boron in aluminum-base alloys. It is hoped that in the coming year these methods can be released for publication.

A method for the determination of zinc in magnesium-base alloys by thiocyanate extraction followed by titration with ethylene diamine tetraacetate has been tested and will be available for early balloting by the committee. The application of the liquid-liquid extraction techniques and the use of chelating agents in general are being considered as to their possible inclusion in many of the analytical methods now under the jurisdiction of this division.

In view of the forthcoming new edition of the Book of ASTM Methods for Chemical Analysis of Metals, a careful review of all methods for analysis of copper-base alloys was undertaken by a task force representative of the industry, and an extensive series of revisions were recommended. It is planned to organize small groups to study these changes and accumulate the necessary data to support the proposed revisions. This program could be completed in time for the new publication. A similar study has also been made on methods for analysis of magnesium-base alloys. It is planned to cover methods for analysis of other non-ferrous metals as rapidly as task forces can be organized and the review and testing program undertaken.

Division G on General Analytical Methods (H. F. Beeghly, chairman).—A Symposium on Solvent Extraction in Metals Analysis was held at the Annual Meeting in Boston, and has been published as Special Technical Publication No. 238. To further enhance the coming symposia, it has been suggested that a special committee or task force be set up for this purpose.

Work continues on the review of methods so as to unify them for the new edition of the Book of ASTM Methods for Chemical Analysis of Metals.

Additional personnel are urgently needed to fulfill activities on the precision and accuracy subdivision.

This report has been submitted to letter ballot of the committee, which consists of 144 members; 91 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

Arba Thomas,
Chairman.

H. Kirtchik,
Secretary.

REPORT OF COMMITTEE E-4
ON
METALLOGRAPHY*

Committee E-4 on Metallography, together with most of its subcommittees, held two meetings during the year: in Boston, Mass., on June 24, 1958, during the Annual Meeting of the Society, and in Pittsburgh, Pa., on February 3, 1959, during ASTM Committee Week.

The committee presently consists of 118 members.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Definitions of:

Terms Relating to Metallography (**E 7 – 55 T**).

Tentative Methods of:

Preparation of Micrographs of Metals and Alloys (Including Recommended Practice for Photography as Applied to Metallography) (**E 2 – 49 T**),

Estimating the Average Grain Size of Wrought Copper and Copper-Base Alloys (**E 79 – 49 T**),

Preparing Quantitative Pole Figures of Metals (**E 81 – 54 T**),

Determining the Orientation of a Metal Crystal (**E 82 – 49 T**), and

Estimating the Average Grain Size of Non-Ferrous Metals, Other Than Copper, and Their Alloys (**E 91 – 51 T**).

Tentative Recommended Practice for:

Thermal Analysis of Metals and Alloys (**E 14 – 51 T**),

Identification of Crystalline Materials by the Hanawalt X-Ray Diffraction Method (**E 43 – 49 T**), and

Dilatometric Analysis of Metallic Materials (**E 80 – 49 T**).

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Selection and Preparation of Samples (W. B. Forgeng, chairman) is currently concerned with a further revision of Methods E 3, and is organizing task groups for each metal specialty area. This program will cover sample preparation for "micro" and "macro" specimens.

Subcommittee III on Nomenclature (P. A. Beck, chairman) has been fostering, through Society Headquarters, the world-wide circulation of the proposed phase nomenclature system. A number of replies have been received and, as more are accumulated, the results will be analyzed for any possible improvements to the proposal.

Subcommittee IV on Photography (G. A. Ellinger, chairman) had, as its principal activity, the sponsorship of the photographic exhibit at the 1958 Annual Meeting. This highly successful venture was conducted by a task group under the leadership of D. I. Finch.

Subcommittee VI on X-Ray Methods (W. L. Fink, chairman) is continuing the revision of the powder pattern techniques, Recommended Practice E 43, and anticipates its conclusion in the near future.

Subcommittee VII on Methods of Thermal Analysis (D. I. Finch, chairman) has prepared a final draft of a recommended practice for temperature *versus* electrical resistance measurements (resistometric determinations).

Subcommittee VIII on Grain Size (R.

*** Sixty-second Annual Meeting of the Society, June 21–26, 1959.**

E. Penrod, chairman) is completing the final grain count checking on new sizes of 0.005, 0.020, 0.150, and 0.200-mm grain sizes for addition to Plate III of Methods E 112. In addition, concentrated effort is being made toward the completion of Plate II of Methods E 112.

Subcommittee IX on Inclusions (Samuel Epstein, chairman) held a most important meeting during ASTM Committee Week, the objective being to ascertain an over-all survey of present-day requirements concerning inclusions and their evaluations. It is evident that the several branches of metallurgical science are concerned with different kinds, amounts, and distributions of inclusions as a function of the production process, material, or product. Thus, in an effort to pinpoint the requirements in several areas, task groups were appointed to study these phases and report their findings at the June meeting.

Subcommittee XI on Electron Microstructure of Metals (N. A. Nielsen, chairman) is sponsoring the Technical Session on Metallography[1] which is being held during the 1959 Annual Meeting.

The subcommittee is currently concerned with reactions in the high-temperature superstrength alloys and in the studies of these and other materials by transmission methods on specially prepared thin foils. Techniques for foil-thinning appear to merit intense investigation.

In addition, the task force concerned with the electron probe microanalysis of metals has several round-robin tests under way, and anticipates detailed comparison of results by the June meeting.

This report has been submitted to letter ballot of the committee, which consists of 118 members; 80 members have returned their ballots, of whom 76 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

MARY R. NORTON,
Secretary.

---

[1] Issued as separate publication *ASTM STP No. 262.*

REPORT OF COMMITTEE E-5
ON
FIRE TESTS OF MATERIALS AND CONSTRUCTION*

Committee E-5 on Fire Tests of Materials and Construction held one meeting during the year: in Pittsburgh, Pa., on February 6, 1959.

The committee consists of 65 members of whom 29 are classified as producers, 7 as consumers, and 29 as general interest, with 2 consulting members.

REVISIONS OF TENTATIVE

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Defining Noncombustibility of Building Materials (E 136 - 58 T):[1]

Title.—Change to read, "Method of Test for Determining Noncombustibility of Elementary Materials."

Section 2.—Delete this section and renumber the subsequent sections accordingly.

Section 3(c).—Change the second sentence to read: "A thermocouple, T_1, may be provided, if desired, in the region of the heating coils for better regulation of the temperature of the air in the furnace space."

Section 4(c).—Change to read: "(*c*) Not less than four identical specimens shall be tested."

Section 5.—In the second line of Paragraph (*a*), place a reference to Footnote 3 after T_2, change the parenthetical phrase to read "(at the approximate position to be occupied by the center of the specimen)," and add the following new Footnote 3:

> [3] This temperature may be measured by means of a thermocouple inserted, during the initial heating period, from the top.

Delete Paragraph (*e*).

Fig. 1.—Revise Fig. 1 relative to location of thermocouple T_2.

Tentative Method of Fire Hazard Classification of Building Materials (E 84 - 50 T):[1] Revise as appended hereto.[2]

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as follows of Standard Methods of Fire Tests of Building Construction and Materials (E 119 - 58):[1]

Section 9.—Change to read as follows:

> 9. *Time of Testing.*—The material or construction shall not be tested until a large proportion of its final strength has been attained, and, if it contains moisture, until the excess has been removed to achieve an air-dry condition as outlined by the following requirements:
>
> (*a*) The air of the seasoning room is to be maintained at a relative humidity of 30 to 35 per cent and a temperature of 70 to 80 F.
>
> (*b*) Specimens are to be exposed to the controlled conditions outlined in Item (*a*) until the interior or dampest section of the assembly attains a 70 per cent relative humidity with a plus and minus tolerance of 5 per cent relative humidity. However, when relative humidity measurements indicate that a specimen has not attained the 70 per cent relative humidity after 12 months of seasoning at the controlled

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

[1] 1958 Book of ASTM Standards, Part 5.

[2] The revised tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 5.

conditions it can be considered to be suitable for fire test.[5]

(c) When there are no facilities for controlling relative humidity and temperature, the requirements for moisture condition outlined in Item (b) should still be followed. Artificial drying may be permitted provided this does not result in abnormal changes in the structural properties of the test specimen. It is recommended that artificial drying temperatures be limited to 120 F.

Add the following new Footnote 5:

[5] A recommended method for determining the relative humidity within a hardened concrete specimen with electric sensing elements is described in Appendix I of a paper by Carl A. Menzel, "A Method for Determining the Moisture Condition of Hardened Concrete in Terms of Relative Humidity," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 1085 (1955); *Bulletin No. D4*, Research and Development Laboratory, Portland Cement Assn., Chicago, Ill. A similar procedure with electric sensing elements can be used to determine the relative humidity within fire test specimens made with other materials.

With wood constructions, the moisture meter based on the electrical resistance method can be used, when appropriate, as an alternate to the relative humidity method to indicate when wood has attained the proper moisture content. Electrical methods are described on pages 320 and 321 of the 1955 edition of the "Wood Handbook" of the Forest Products Laboratory, U. S. Department of Agriculture. The relationships between relative humidity and moisture content are given by the graphs in Fig. 23 on p. 327. They indicate that wood has a moisture content of 13 per cent at a relative humidity of 70 per cent for a temperature of 70 to 80 F.

New Section.—Add a new Section 30 to read as follows, renumbering the present Section 30 and subsequent sections accordingly:

30. *Application.*—This test procedure is to be used for classification of ceilings which are not an integral part of a floor construction and where 36 in. or more of space is provided above the top of the joists or beams supporting and protected by the ceiling.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Fire Tests of Materials and Construction (A. J. Steiner, chairman) has made important recommendations for changes in Methods E 119 which are given in this report. Other matters requiring further study deal with (1) a more exact method of determining time of load failure of floors under fire exposure, (2) the need of long-span furnaces for testing beams, and (3) the acquirement and proving of surface thermocouple protection pads to replace the one described in Section 4(a) which is no longer obtainable.

Subcommittee III on Fire Tests of Wall Opening Assemblies (H. D. Foster, chairman) has prepared a working draft of a method of fire tests of window assemblies and has under consideration refinements to be made preparatory to submitting it as a proposed tentative.

Subcommittee IV on Fire Tests of Acoustical and Similar Finishes (C. H. Yuill, chairman) has revised the flame-spread test method (E 84) which covers the large tunnel furnace of which there are six in the United States and one in Canada. It has a task group working on minor editorial revisions of the test method and on the correlation of results of tests made in a number of furnaces. A separate task group is studying the results of tests made in the smaller tunnel furnace developed by the Forest Products Laboratories and in the radiant panel furnace sponsored by the National Bureau of Standards. Test methods based on these two types of furnaces have been prepared and are being considered either as alternates to

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Method E 84 or as methods for use in research and development work.

Subcommittee V on Nomenclature and Definitions (J. V. Ryan, chairman) has proposed the revisions of Method E 136 given in this report. It has under consideration definitions for the basic terms used in connection with fire testing and fire test ratings.

Subcommittee X on Research (A. F. Robertson, chairman) is not responsible for test methods. It has made a critical review of the entire field of fire testing and will make recommendations relative to the need of new or revised test methods.

This report has been submitted to letter ballot of the committee, which consists of 65 voting members; 46 members returned their ballots, of whom 44 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committeee,

W. J. KREFELD,
Chairman.

H. D. FOSTER,
Secretary.

REPORT OF COMMITTEE E-6
ON
METHODS OF TESTING BUILDING CONSTRUCTIONS*

Committee E-6 on Methods of Testing Building Constructions has met twice during the past year: in Boston, Mass., during the Annual Meeting of the Society, and at Pittsburgh, Pa., during ASTM Committee Week.

At the Spring Meeting in Pittsburgh, Subcommittee VIII on Windows sponsored a Symposium on Testing Window Assemblies.[1]

The Committee consists of 99 members, of whom 77 are voting members; 32 are classified as producers, 7 as consumers, and 38 as general interest members. There are 7 consulting members.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Panels for Light-Building Construction (J. A. Liska, chairman) is conducting investigations in two problem areas: (*a*) a standard method for the preparation of test panels for the wet-racking test, and (*b*) the lateral nail-holding properties of sheathing materials whenever racking tests are made. Progress is being made on both problems.

Subcommittee II on Durability (E. C. Shuman, chairman) reports that, while much is known about the factors that affect durability, the information does not appear to be in the hands of those who should be using it—architects, in particular. No recommendations are being made at this time.

Subcommittee III on Sound Transmission (R. K. Cook, chairman) explained the existence of other standards (ISO and ASA) in this field that were published subsequent to the ASTM standard; certain members of this subcommittee helped write the ASA standard which reflects more recent thinking than ASTM Recommended Practice E 90. It is felt that the ISO standard involves unnecessarily large panel sizes which make possible accurate results below the frequencies considered important. There is no agreement on how to arrive at a single value representing wall performance; it is agreed that a reliable method of field testing transmission loss for a given wall does not exist. A task group will draft a revision of Recommended Practice E 90, which will then be voted upon by the subcommittee.

Subcommittee IV on Unit Masonry (H. C. Plummer, chairman) is revising a method of test for bond strength of mortar to masonry units. A task group has been formed to investigate test methods for shear and diagonal tension.

Subcommittee V on Light Trusses (D. E. Kennedy, chairman) has prepared two drafts of a method of testing wooden trussed rafters. The second draft was discussed at a meeting of the subcommittee in Pittsburgh in February and several changes were agreed upon. It was also decided to review the question of duplication between this proposed standard and the existing Methods E 73.

Subcommittee VI on Load Tests on Completed Structures (W. R. Schriever, chairman) made the preparation of a method for load tests on floors its first assignment. Two drafts were prepared and the method was extended to include flat and low slope roofs. A danger of overlapping with Methods E 72 – 55 was discussed at the recent Pittsburgh meeting, and it was decided to set up a task force with Subcommittees I and V to look into this.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Issued as separate publication *ASTM STP No. 251.*

Subcommittee VII on Vapor Barriers Beneath Concrete Slabs on Ground (R. F. Luxford, chairman) is balloting the Suggested Methods of Testing Materials Used as Vapor Barriers Under Concrete Slabs and as Ground Cover in Crawl Spaces.

Subcommittee VIII on Windows (R. B. Crepps, chairman) has started a program to evaluate and prepare standard test methods of window assemblies for such items as structural behavior, air infiltration, water leakage and, perhaps, thermal performance. Methods and procedures now in use by commercial or university laboratories and manufacturers of windows will be collected and analyzed. Mr. R. B. Crepps, Structural Engineering Department, Purdue University, West Lafayette, Ind., welcomes information from, and contact with, all groups either having, using, or contemplating test equipment in the fields mentioned.

This report has been submitted to letter ballot of the committee; 59 members have returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. F. LEGGET,
Chairman.

R. A. BIGGS,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-6 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of:

Test for Bond Strength of Mortar to Masonry Units (E 149 – 59 T), and

Testing Materials Used as Vapor Barriers Under Concrete Slabs and as Ground Cover in Crawl Spaces (E 154 – 59 T).

Tentative Method E 149 was accepted by the Standards Committee on September 15, 1959, and Tentative Methods E 154 were accepted on October 27, 1959. The methods appear in the 1959 Supplement to Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE E-7
ON
NONDESTRUCTIVE TESTING*

Committee E-7 on Nondestructive Testing met in Boston, Mass., on June 26, 1958, in conjunction with the Annual Meeting of the Society. Six subcommittees, the Executive Council, and several task groups also met at that time. Another series of meetings was held in Pittsburgh, Pa., in February, 1959, during ASTM Committee Week, including the first meeting of Subcommittee VII on Electromagnetic Methods.

During the year Sidney Low, Chapman Valve Mfg. Co., was appointed chairman of Subcommittee II on Reference Radiographs, and J. W. Allen, Oak Ridge National Laboratory, as chairman of Subcommittee VII on Electromagnetic Methods.

The committee is sponsoring an all-day Symposium on Nondestructive Testing[1] during the Third Pacific Area National Meeting in San Francisco next October. S. A. Wenk, Applied Radiation Corp., is chairman of the symposium committee.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Tentative Method for Controlling Quality of Radiographic Testing as appended hereto.[2] This is a revision of the proposed Tentative Method for Quality Inspection by Radiographic Testing appended to the 1958 Preprinted Report of Committee E-7. The document was withdrawn last year to give further consideration to negative viewpoints.

The letter ballot results on the attached proposal were 76 affirmative, 5 negative, and 34 not voting. After intensive effort and many discussions of the negative votes, the Executive Council of Committee E-7 feels that the attached proposal represents the best compromise possible. This same work has been the basis for MIL specifications—one of which makes use of the penetrameter described in the ASTM document—mandatory for all applicable radiographic inspection.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Radiographic Recommended Practice (G. R. Forrer, chairman) is evaluating radiographs exposed at varying distances from the radiation source, for changes in image appearance. Also under way is a revision of the Recommended Practice for Radiographic Testing (E 94 – 52 T).

Subcommittee II on Reference Radiographs (S. Low, chairman) has two very active tasks groups developing additional reference radiographs.

The Task Group on Reference Radiographs for Heavy Steel Castings (F. H. Allison, Jr., chairman) is developing reference radiographs for heavy steel castings in 3-in., 6-in., and 12-in. sections. The ASTM Administrative Committee on Research has granted $2200 to help in this work.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] To be issued as separate publication *ASTM STP No. 278.*

[2] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 3.

The Task Group on Reference Radiographs for Aluminum and Magnesium Castings (J. Battema, chairman) is in the final stages of preparing an album of reference radiographs to be sponsored jointly by ASTM and the Aircraft Industries Assn. These radiographs will be reproduced directly by radiographic means rather than the photographic reproductions now used for Reference Radiographs E 98 – 53 T.

Another task group has been appointed to study the problem of brazed honeycomb construction with the aim of preparing a document including terminology, reference radiographs, etc. This task group is under the chairmanship of J. Fenton, Convair.

Note has been taken of the reference radiographs for bronze castings issued by the U. S. Navy Bureau of Ships. The subcommittee is determining whether ASTM can in any way aid in supplementing this set of radiographs and expediting its use. The past chairman of Subcommittee II, Noah Kahn, was deeply involved in this project.

Subcommittee III on Magnetic Particle and Penetrant Testing (H. Migel, chairman) has reviewed a preliminary draft of a method for liquid penetrant inspection employing fluorescent and color contrast procedures. The results of the discussion will be incorporated in another draft.

The subcommittee is investigating the possibility of a set of photographs illustrating discontinuities revealed by penetrant inspection.

Subcommittee IV on Nomenclature and Definitions is being reorganized by C. H. Hastings at the suggestion of S. A. Wenk. It will be engaged in a revision of the Industrial Radiographic Terminology for Use in Radiographic Inspection of Castings and Weldments (E 52 – 49 T). The new reference radiographs being prepared indicate that E 52 is not up to date.

Subcommittee V on Radiographic Procedure (C. H. Hastings, chairman) prepared the proposed Tentative Method for Controlling Quality of Radiographic Testing as appended to this report.[2]

Subcommittee VI on Ultrasonic Testing Procedure (J. C. Smack, chairman) is in the final stages of developing recommended practices for ultrasonic inspection of ferrous welds and for ultrasonic testing by the immersion method. Other task groups are developing a glossary of terms, a recommended practice for ultrasonic inspection using angle projection of beam by surface contact, and reference blocks for ultrasonic testing of steel. One other task group has just begun work on a practice for standardization of ultrasonic instruments and transducers.

Subcommittee VII on Electromagnetic Methods (J. W. Allen, chairman) held its first meeting on February 5, 1959, during ASTM Committee Week in Pittsburgh. The attendance was excellent, with many representatives of companies producing non-ferrous products present. Task groups were appointed to investigate various types of nondestructive tests conducted by electromagnetic methods.

Respectfully submitted on behalf of the committee,

J. H. BLY,
Chairman.

A. GOBUS,
Secretary.

REPORT OF COMMITTEE E-9
ON
FATIGUE

Committee E-9 on Fatigue held two meetings during the year: on June 24, 1958, at Boston, Mass., and on February 2, 1959, at Pittsburgh, Pa.

During the above period five members were elected to the main E-9 Committee. C. E. Phillips, MERL, East Kilbride, Scotland, was added to the group of corresponding members. The committee lost one of its most active members in the death of Sam Gordon of Battelle Memorial Inst.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (T. J. Dolan, chairman).—The symposium presented at Boston on "Basic Mechanisms of Fatigue" has been published as *ASTM STP No. 237.* In the proposals being made for the formation of a new ASTM division on materials sciences, the above publication was cited as an example of the society's interest in fundamental knowledge in the materials field. Subcommittee I also arranged for L. F. Coffin as a speaker at the Committee E-9 meeting at Boston. Mr. Coffin summarized knowledge concerning low-cycle fatigue.[1]

Subcommittee II on Papers (D. G. Richards, chairman).—Sufficient papers are under review to warrant two sessions at Atlantic City, N. J., in June 1959, and two sessions at San Francisco, Calif., in October 1959.

Subcommittee III on Survey (W. S. Hyler, chairman).—During the year, *ASTM STP No. 9-I,* "References on Fatigue 1957," was issued. Work is progressing on a 10-year index. A survey of current projects was published in the January 1959 issue of the ASTM BULLETIN.

Subcommittee IV on Large Machines and Test Correlation (J. F. Millan, chairman).—Papers are being solicited with the objective of a symposium in 1960.

Subcommittee V on Aircraft Structural Fatigue Problems (H. F. Hardrath, chairman).—There will be three sessions on fatigue[2] at the Third Pacific Area National Meeting to be held October 11 to 16, 1959, at San Francisco. The aircraft companies have been solicited for papers and the response has been such that all of the sessions are being devoted to aircraft problems.

Task Force on Definitions and Symbols (G. R. Gohn, chairman).—The task force has completed the Definitions and Symbols for Fatigue Testing which appears as an appendix to this report.[3] Eventually it will be utilized as a revision of Section II of *ASTM STP No. 91,* "Manual on Fatigue." A major change is the incorporation of statistical terms.

This report has been submitted to letter ballot of the committee, which consists of 51 members; 21 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

O. J. HORGER,
Secretary.

---

[1] L. F. Coffin, Jr., "A Mechanism for Nonpropagating Fatigue Cracks," *Proceedings,* Am. Soc. Testing Mats., Vol. 58, p. 570 (1958).

[2] To be issued as separate publication *ASTM STP No. 274.*

[3] See p. 596.

APPENDIX

PROPOSED REVISION OF SECTION II, MANUAL ON FATIGUE TESTING, STP 91

This proposed revision is intended to replace Section II of the Manual on Fatigue Testing, *STP 91*. It will also supersede the definitions now published in A Tentative Guide for Fatigue Testing and the Statistical Analysis of Fatigue Data (Supplement to Manual on Fatigue Testing), *STP 91-A*.

SECTION II.—DEFINITIONS AND SYMBOLS FOR FATIGUE TESTING[1]

Scope

This section covers the principal terms and symbols relating to fatigue testing and the statistical analysis of fatigue data. The general definitions are restricted and interpreted, when necessary, to make them particularly applicable and practicable for use in the other sections of the Manual on Fatigue Testing,[2] and in technical articles intended for publication by the Society. To encourage uniformity of terminology throughout the Society, the terms dealing primarily with fatigue testing and test methods are also published in the Definitions of Terms Relating to Methods of Mechanical Testing (ASTM Designation: E 6).[3] The symbols used are, in general, those recommended in the American Standard Letter Symbols for Mechanics of Solid Bodies (ASA No. Z10.3–1948). For stress, the use of S with appropriate lower case subscripts is preferred for general purposes; for mathematical analysis, the use of Greek symbols is generally preferred.

Index of Terms

The definitions appear in the following subsections:

A. Fatigue Tests and Test Methods

Term	Subsection
Alternating Stress Amplitude	11
Constant Lifetime Fatigue Diagram	16
Cycle Ratio	25
Cycle, Stress	12
Diagram, Constant Lifetime Fatigue	16
Diagram, *S-N*	15
Ellipse Relation	21
Fatigue	1
Fatigue Diagram, Constant Lifetime	16
Fatigue Life	2
Fatigue Limit	4
Fatigue Notch Factor	23
Fatigue Notch Sensitivity	24
Fatigue Ratio	5
Fatigue Strength at *N* Cycles	3
Gerber Relation	20
Goodman Relation	17
Goodman Relation, Modified	18
Haigh-Soderberg Relation	19
Level, Stress	3, Note 5
Life, Fatigue	2
Limit, Fatigue	4
Maximum Stress	7
Mean Stress	9
Minimum Stress	8
Nominal Stress	6
Notch Factor, Fatigue	23
Notch Sensitivity, Fatigue	24
Range of Stress	10
Ratio, Cycle	25

---

[1] Drafted by a Task Group consisting of H. J. Grover, Battelle Memorial Institute; H. F. Hardrath, National Aeronautics and Space Administration; E. H. Schuette, Dow Chemical Co.; and G. R. Gohn (chairman), Bell Telephone Laboratories, Inc.

[2] Issued as separate publication *ASTM STP No. 91*.

[3] 1958 Book of ASTM Standards, Part 3.

Term	Subsection
Ratio, Fatigue	5
Ratio, Stress	14
Relation, Ellipse	21
Relation, Gerber	20
Relation, Goodman	17
Relation, Haigh-Soderberg	19
Relation, Modified Goodman	18
Relation, Soderberg (or Haigh-Soderberg)	19
S-N Diagram	15
Soderberg (or Haigh-Soderberg) Relation	19
Steady Stress Component	9
Strength at *N* Cycles, Fatigue	3
Stress Amplitude, Alternating	11
Stress Component, Steady	9
Stress Component, Variable	11
Stress Concentration Factor, Theoretical	22
Stress Cycle	12
Stress Cycles Endured	13
Stress Level	3, Note 5
Stress, Maximum	7
Stress, Mean	9
Stress, Minimum	8
Stress, Nominal	6
Stress, Range of	10
Stress Ratio	14
Theoretical Stress Concentration Factor	22
Variable Stress Component	11

B. STATISTICAL ANALYSIS OF FATIGUE DATA

Term	Subsection
Arithmetic Mean	43
Average, Sample	43
Coefficient, Confidence	50
Confidence Coefficient	50
Confidence Interval	48
Confidence Level	50
Confidence Limits	49
Curve for 50% Survival, *S-N*	31
Curve for *N* Cycles, Response	33
Curve for *p*% Survival, *S-N*	32
Deviation, Sample Standard	45
Distribution, Frequency	36
Estimate	40
Estimate, Interval	47
Estimate, Point	41
Estimation	39
Fatigue Life for *p*% Survival	27
Fatigue Life, Median	26
Fatigue Limit for *p*% Survival	30
Fatigue Strength at *N* Cycles, Median	28
Fatigue Strength for *p*% Survival at *N* Cycles	29
Frequency Distribution	36
Interval, Confidence	48
Interval, Estimate	47
Interval, Tolerance	51
Level, Confidence	50
Level, Significance	57
Level, Tolerance	53
Life, Fatigue for *p*% Survival at *N* Cycles	29
Life, Median Fatigue	26
Life at *N* Cycles, Median Fatigue	28
Life for *p*% Survival, Fatigue	27
Limit for *p*% Survival, Fatigue	30
Limits, Confidence	49
Limits, Tolerance	52
Mean, Arithmetic	43
Median Fatigue Life	26
Median Fatigue Strength at *N* Cycles	28
Median, Sample	42
Parameter	37
Percentage, Sample	46
Point Estimate	41
Population	34
Response Curve for *N* Cycles	33
S-N Curve for 50% Survival	31
S-N Curve for *p*% Survival	32
Sample	35
Sample Average	43
Sample Median	42
Sample Percentage	46
Sample Standard Deviation	45
Sample Variance	44
Significance Level	57
Significance, Test of	56
Significant	54
Standard Deviation, Sample	45
Statistic	38
Statistic, Test-	55
Test of Significance	56
Test-Statistic	55
Tolerance Interval	51
Tolerance Level	53
Tolerance Limits	52
Variance, Sample	44

A. DEFINITIONS RELATING TO FATIGUE TESTS AND TEST METHODS

1. **Fatigue.**—The phenomenon of fracture under repeated or fluctuating stress (Note 1) having a maximum value generally less than the tensile strength of the material.

NOTE 1.—Fatigue may also be expressed in terms of repeated or fluctuating loads or deformations. The latter is especially useful in describing fatigue under conditions where the elastic limit of the material is exceeded. (Deformation is the sum of elastic and plastic strains.) In the definitions that follow, the terms "repeated stress," "cyclic stress," or "fluctuating stress" are intended to cover any of the three conditions, that is, stress, load, or strain.

2. **Fatigue Life,** *N*.—The number of stress cycles (Note 2) that a specimen sustains before failure (Note 3) under a

given test condition and criterion of failure.

NOTE 2.—In some instances this may be either load or strain cycles (see definition for Fatigue).

NOTE 3.—The criterion of failure should be defined to avoid ambiguity.

3. **Fatigue Strength** (Note 4) **at** N **Cycles,** S_N.—The hypothetical value of the median stress level (Note 5) at which a specimen would have a life of exactly N cycles (also see Section 28).

NOTE 4.—"Fatigue Strength" may also be considered to be a preferred general term of which "fatigue limit" is a special case.

NOTE 5.—In the uniaxial fatigue test, the term stress-level is usually applied to the peak or maximum stress in the cycle. Multiaxial stress requires a more rigorous definition which is beyond the intended scope of this section.

NOTE 6.—For a preassigned cycle life, N, this value is rarely observed as an actual result of fatigue testing.

NOTE 7.—If the stress is not completely reversed, the fatigue strength may be expressed in terms of the alternating stress amplitude of the maximum stress; whichever method is used, it is also necessary to state the value of the mean stress, minimum stress, or stress ratio.

4. **Fatigue Limit,** S_f.—The limiting value of the median fatigue strength as N becomes very large.

NOTE 8.—If the stress is not completely reversed, the fatigue limit may be expressed in terms of the alternating stress amplitude or the maximum stress; whichever method is used, it is also necessary to state the value of the mean stress, minimum stress, or stress ratio.

NOTE 9.—Certain materials and environments preclude the attainment of a fatigue limit. Values tabulated as "fatigue limits" in the literature are generally (but not always) median fatigue strengths at some specified cycle life.

5. **Fatigue Ratio.**—The ratio of the fatigue limit, S_f, or fatigue strength, S_N, to the static tensile strength, S_u, that is,

$$\frac{S_f}{S_u} \text{ or } \frac{S_N}{S_u}$$

NOTE 10.—This definition is applicable only for completely reversed stress and long life times.

6. **Nominal Stress,** S.—The stress calculated on the net section by simple theory such as

$$S = \frac{P}{A} \quad \text{or} \quad S = \frac{Mc}{I} \quad \text{or} \quad S_s = \frac{Tc}{J}$$

without taking into account the variations in stress conditions caused by geometrical discontinuities such as holes, grooves, fillets, etc.

7. **Maximum Stress,** S_{max}.—The highest algebraic value of the nominal stress in the stress cycle, tensile stress being considered positive and compressive stress negative.

8. **Minimum Stress,** S_{min}.—The lowest algebraic value of the nominal stress in the stress cycle, tensile stress being considered positive and compressive stress negative.

9. **Mean Stress (or Steady Stress Component),** S_m.—The algebraic mean of the maximum and minimum nominal stress in one cycle, that is,

$$S_m = \frac{(S_{max} + S_{min})}{2}$$

10. **Range of Stress,** S_r.—The algebraic difference between the maximum and minimum nominal stress in one cycle, that is,

$$S_r = S_{max} - S_{min}$$

11. **Alternating Stress Amplitude** (or **Variable Stress Component),** S_a.—One half the range of stress, that is,

$$S_a = \frac{S_r}{2} = \frac{S_{max} - S_{min}}{2}$$

12. **Stress Cycle.**—For constant amplitude testing, a stress cycle is the smallest section of the stress-time function which is repeated periodically. For more complex testing conditions the

"stress cycle" generally requires unique definition.

13. **Stress Cycles Endured, *n*.**—The number of cycles which a specimen has endured at a given stress level at any stage of a fatigue test.

14. **Stress Ratio, *R or A*.**—The algebraic ratio of two definitive stress components in a stress cycle.

R is the ratio of the minimum to the maximum stress, that is,

$$R = \frac{S_{min}}{S_{max}}$$

A is the ratio of the alternating stress amplitude to the mean stress, that is,

$$A = \frac{S_a}{S_m}$$

NOTE 11.—It develops from these definitions that

$$R = \frac{(1 - A)}{(1 + A)}, \text{ and } A = \frac{(1 - R)}{(1 + R)}.$$

15. ***S-N* Diagram.**—A plot of stress against number of cycles to failure. For *N*, a log scale is almost always used. For *S*, a linear scale is used most often, but a log scale is sometimes used.

16. **Constant Lifetime Fatigue Diagram.**—A plot (usually on rectangular coordinates) of a family of curves, each of which represents a single constant lifetime relating the variable stress (that is, the alternating stress amplitude, the maximum stress, or the minimum stress) and the mean stress. The constant lifetime fatigue diagram is generally derived from an *S-N* diagram containing a family of *S-N* curves, each of which represents a different stress ratio.

NOTE 12.—The Goodman, Modified Goodman, Soderberg (or Haigh-Soderberg), Gerber, and Ellipse relations defined in Subsections 17 to 21 may be portrayed by constant lifetime diagrams, provided the proper set of coordinate axes is selected. However, these relations generally express the *S-N* relationship.

17. **Goodman Relation.**—An empirical formula relating the fatigue limit (expressed in terms of the alternating stress amplitude for a given mean stress) to the mean stress and the ultimate tensile strength (S_u). It may be expressed by the equation:

$$S_a = \frac{S_u}{3}\left(1 - \frac{S_m}{S_u}\right)$$

NOTE 13.—Goodman assumed that the cyclic stress superimposed on the minimum stress was suddenly applied and, on this false assumption, it developed that the fatigue limit for $R = 0$ was $\frac{S_u}{2}$ and the fatigue limit for $R = -1$ was $\frac{S_u}{3}$, a condition which is approximated only in a few of the low carbon steels.

NOTE 14.—The Goodman relation is frequently expressed in terms of S_{max} and S_{min} rather than S_a. Thus

$$\frac{S_{max} - S_{min}}{2} = \frac{S_u}{3}\left(1 - \frac{S_m}{S_u}\right)$$

18. **Modified Goodman Relation.**—An empirical formula relating the fatigue limit (expressed in terms of the alternating stress amplitude for a given mean stress) to the fatigue limit for zero mean stress, the mean stress, and the ultimate tensile strength (S_u). It may be expressed by the equation:

$$S_a = S_f\left(1 - \frac{S_m}{S_u}\right)$$

NOTE 15.—In the more general form, the fatigue strength at *N* cycles may be substituted for the fatigue limit thus giving rise to the equation:

$$S_a = S_N\left(1 - \frac{S_m}{S_u}\right)$$

NOTE 16.—The general relation is frequently expressed in terms of S_{max} and S_{min} rather than S_a. Thus

$$\frac{S_{max} - S_{min}}{2} = S_N\left(1 - \frac{S_m}{S_u}\right)$$

19. **Soderberg (or Haigh-Soderberg) Relation.**—An empirical formula relating the fatigue limit (expressed in terms of

the alternating stress amplitude for a given mean stress) to the fatigue limit for zero mean stress, the mean stress, and the tensile yield strength (S_{ty}). It may be expressed by the equation:

$$S_a = S_f \left(1 - \frac{S_m}{S_{ty}}\right)$$

NOTE 17.—In the more general form, the fatigue strength at N cycles may be substituted for the fatigue limit thus giving rise to the equation:

$$S_a = S_N \left(1 - \frac{S_m}{S_{ty}}\right)$$

NOTE 18.—The general relation is frequently expressed in terms of S_{max} and S_{min} rather than S_a. Thus

$$\frac{S_{max} - S_{min}}{2} = S_N \left(1 - \frac{S_n}{S_{ty}}\right)$$

20. **Gerber Relation.**—An empirical formula relating the fatigue strength (expressed in terms of the alternating stress amplitude for a given mean stress) to the fatigue limit at zero mean stress, the mean stress, and the ultimate tensile strength (S_u). It may be expressed by the equation:

$$S_a = S_f \left[1 - \left(\frac{S_m}{S_u}\right)^2\right]$$

NOTE 19.—In the more general form, the fatigue strength at N cycles may be substituted for the fatigue limit thus giving rise to the equation:

$$S_a = S_N \left[1 - \left(\frac{S_m}{S_u}\right)^2\right]$$

NOTE 20.—The general relation is frequently expressed in terms of S_{max} and S_{min} rather than S_a. Thus

$$\frac{S_{max} - S_{min}}{2} = S_N \left[1 - \left(\frac{S_m}{S_u}\right)^2\right]$$

21. **Ellipse Relation.**—An empirical formula relating the fatigue limit (expressed in terms of the alternating stress amplitude for a given mean stress) to the fatigue limit at zero mean stress, the mean stress, and the ultimate tensile strength (S_u). It may be expressed by the equation:

$$S_a = S_f \sqrt{1 - \left(\frac{S_m}{S_u}\right)^2}$$

NOTE 21.—In the more general form, the fatigue strength at N cycles may be substituted for the fatigue limit thus giving rise to the equation:

$$S_a = S_N \sqrt{1 - \left(\frac{S_m}{S_u}\right)^2}$$

NOTE 22.—The general relation is frequently expressed in terms of S_{max} and S_{min} rather than S_a. Thus

$$\frac{S_{max} - S_{min}}{2} = S_N \sqrt{1 - \left(\frac{S_m}{S_u}\right)^2}$$

22. **Theoretical Stress Concentration Factor,** or **Stress Concentration Factor,** K_t.—The ratio of the greatest stress in the region of the notch or other stress concentrator as determined by the theory of elasticity (or by experimental procedures that give equivalent values) to the corresponding nominal stress.

NOTE 23.—The theory of plasticity should not be used to determine a theoretical stress concentration factor.

23. **Fatigue Notch Factor** (Note 24), K_f.—The ratio of the fatigue strength of a member or specimen with no stress concentration to the fatigue strength at the same number of cycles with stress concentration.

NOTE 24.—This has sometimes been called "Fatigue Strength Reduction Factor," a term that is no longer recommended.

NOTE 25.—K_f is ambiguous unless: (*1*) the method of computation is described; (*2*) the geometry and size of the member or specimens are given; (*3*) the pertinent stress levels and life times are stated. Generally the life times are expressed in terms of the limiting values rather than at some intermediate value of time.

24. **Fatigue Notch Sensitivity,** Q.—A measure of the degree of agreement between K_f and K_t for a particular speci-

men or member of given size and material containing a stress concentrator of given size and shape.

NOTE 26.—One definition of fatigue notch sensitivity that has widespread use is $q = \frac{(K_f - 1)}{(K_t - 1)}$. For this definition fatigue notch sensitivity can vary between zero (where $K_f = 1$) and unity (where $K_f = K_t$).

25. **Cycle Ratio,** *C*.—The ratio of the number of stress cycles (n) applied at a given stress level to the expected fatigue life (N) at that stress level based on the *S-N* diagram, that is, $C = n/N$.

B. DEFINITIONS RELATING TO STATISTICAL ANALYSIS OF FATIGUE DATA

26. **Median Fatigue Life.**—The middlemost of the observed fatigue life values of the individual specimens in a group tested under identical conditions.

NOTE 27.—The use of the sample median instead of the arithmetic mean (that is, the average) is preferred.

NOTE 28.—In the literature, the abbreviated term "fatigue life" usually has meant the median fatigue life of the group. However, when applied to a collection of data without further qualification the term "fatigue life" is ambiguous.

27. **Fatigue Life for** ***p*% Survival.**—An estimate of the fatigue life that *p*% of the population would attain or exceed at a given stress level. The observed value of the median fatigue life estimates the fatigue life for 50% survival. Fatigue life for *p*% survival values, where *p* is any number, such as, 95, 90, etc., may also be estimated from the individual fatigue life values.

28. **Median Fatigue Strength at** ***N*** **Cycles.**—An estimate of the stress level at which 50% of the population would survive *N* cycles.

NOTE 29.—The estimate of the median fatigue strength is derived from a particular point of the fatigue life distribution, since there is no test procedure by which a frequency distribution of fatigue strength at *N* cycles can be directly observed.

NOTE 30.—This is a special case of the more general definition (see subsection 29).

29. **Fatigue Strength for** ***p*% Survival at** ***N*** **Cycles.**—An estimate of the stress level at which *p*% of the population would survive *N* cycles; *p* may be any number, such as, 95, 90, etc.

NOTE 31.—The estimates of the fatigue strength for *p*% survival values are derived from particular points of the fatigue life distribution, since there is no test procedure by which a frequency distribution of fatigue strengths at *N* cycles can be directly observed.

30. **Fatigue Limit for** ***p*% Survival.**—The limiting value of fatigue strength for *p*% survival as *N* becomes very large; *p* may be any number, such as, 95, 90, etc.

NOTE 32.—See Notes 8 and 9 under definition for **Fatigue Limit**.

31. ***S-N*** **Curve for 50% Survival.**—A curve fitted to the median values of fatigue life at each of several stress levels. It is an estimate of the relationship between applied stress and the number of cycles-to-failure that 50% of the population would survive.

NOTE 33.—This is a special case of the more general definition (see subsection 32).

NOTE 34.—In the literature, the abbreviated term "*S-N* Curve" usually has meant either the *S-N* curve drawn through the means (averages) or the medians (50% values) for the fatigue life values. Since the term "*S-N* Curve" is ambiguous, it should be used in technical papers only when adequately described.

32. ***S-N*** **Curve for** ***p*% Survival.**—A curve fitted to the fatigue life of *p*% survival values at each of several stress levels. It is an estimate of the relationship between applied stress and the number of cycles-to-failure that *p*% of the population would survive. *p* may be any number, such as, 95, 90, etc.

NOTE 35.—Caution should be used in drawing conclusions from extrapolated portions of the

S-N curves. In general, the *S-N* curves should not be extrapolated beyond observed life values.

33. **Response Curve for N Cycles.**—A curve fitted to observed values of percentage survival at N cycles for each of several stress levels, where N is a preassigned number such as 10^6, 10^7, etc. It is an estimate of the relationship between applied stress and the percentage of the population that would survive N cycles.

NOTE 36.—Values of the median fatigue strength at N cycles and the fatigue strength for $p\%$ survival at N cycles may be derived from the response curve for N cycles if p falls within the range of the per cent survival values actually observed.

NOTE 37.—Caution should be used in drawing conclusions from extrapolated portions of the response curves. In general, the curves should not be extrapolated to other values of p.

34. **Population.** — The hypothetical collection of all possible test specimens that could be prepared in the specified way from the material under consideration.

35. **Sample.**—The specimens selected from the population for test purposes.

NOTE 38.—The method of selecting the sample determines the population about which statistical inference or generalization can be made.

36. **Frequency Distribution.**—The way in which the frequencies of occurrence of members of a population, or a sample, are distributed according to the values of the variable under consideration.

37. **Parameter.**—A constant (usually unknown) defining some property of the frequency distribution of a population, such as, a population median or a population standard deviation.

38. **Statistic.**—A summary value calculated from the observed values in a sample.

39. **Estimation.**—A procedure for making a statistical inference about the numerical values of one or more unknown population parameters from the observed values in a sample.

40. **Estimate.**—The particular value, or values for a given sample computed by an estimation procedure.

41. **Point Estimate.**—The estimate given by a single statistic.

42. **Sample Median.**—The middle value when all observed values in a sample are arranged in order of magnitude. If the sample size is even, it is the average of the two middle-most values. It is a point estimate of the population median, or 50% point.

43. **Sample Average (Arithmetic Mean).**—The sum of all the observed values in a sample divided by the sample size. It is a point estimate of the population mean.

44. **Sample Variance.**—The sum of the squares of the differences between each observed value and the sample average divided by the sample size minus one. It is a point estimate of the population variance.

NOTE 39.—The term sample variance may also have other meanings in different fields.

45. **Sample Standard Deviation.**—The square root of the sample variance. It is a point estimate of the population standard deviation, a measure of the "spread" of the frequency distribution of a population.

46. **Sample Percentage.**—The percentage of observed values between two stated values of the variable under consideration. It is a point estimate of the percentage of the population between the same two stated values. (One stated value may be $-\infty$ or $+\infty$.)

47. **Interval Estimate.**—The estimate given by two statistics, defining the end points of an interval.

48. **Confidence Interval.**—An interval estimate (a computed band) of a population parameter computed so that the statement "the population parameter lies in this interval" will be true, on the average, in a stated proportion of the times such statements are made.

49. **Confidence Limits.**—The two statistics that define a confidence interval.

50. **Confidence Level (or Coefficient).**—The stated proportion of the times the confidence interval is expected to include the population parameter.

51. **Tolerance Interval.**—An interval computed so that it will include at least a stated percentage of the population with a stated probability.

52. **Tolerance Limits.**—The two statistics that define a tolerance interval. (One value may be $-\infty$ or $+\infty$.)

53. **Tolerance Level.**—The stated probability that the tolerance interval includes at least the stated percentage of the population. It is not the same as a confidence level but the term confidence level is frequently associated with tolerance intervals.

54. **Significant.**—Statistically meaningful. (An effect or difference between populations is said to be present if the value of a test-statistic is significant, that is, lies outside of predetermined limits.)

NOTE 40.—An effect which is statistically meaningful may or may not have engineering significance.

55. **Test-Statistic.**—A function of the observed values in a sample that is used in a test of significance.

56. **Test of Significance.**—A test of the hypothesis that the effect is not present. The rejection of the hypothesis indicates that the effect is present.

57. **Significance Level.**—The stated probability (risk) that a given test of significance will reject the hypothesis that a specified effect is not present when the hypothesis is true.

APPENDIX

LIST OF SYMBOLS USED IN FATIGUE

The following terms are frequently used in lieu of or along with the terms covered by the preceding definitions. In general these symbols are those recommended in the American Standard Letter Symbols for Mechanics of Solid Bodies (ASA No. Z10.3—1948). For stress, the use of *S* with appropriate lower case subscripts is preferred for general purposes; for mathematical analysis the use of Greek symbols is generally preferred.

(Symbol)	Term
A	Area of cross-section, Stress ratio
C	Cycle ratio
c	Distance from centroid to outermost fiber
D or d	Diameter
E	Modulus of elasticity
ϵ	Strain
G	Modulus of elasticity in shear
γ	Shear strain
I	Moment of inertia
i	Subscript denoting i^{th} term
J	Polar moment of inertia
K_f	Fatigue notch factor
K_t	Theoretical stress concentration factor
M	Bending moment
N	Fatigue life (number of cycles)
n	Number of cycles endured, Sample size
P	Load
p	Probability of failure, Probability of survival
q	Fatigue notch sensitivity
R	Stress ratio
S (or σ)	Nominal stress
S_a	Alternating stress amplitude
S_c	Nominal compressive stress
S_f	Fatigue limit
S_{cy}	Compressive yield strength
S_m	Mean stress
S_{max}	Maximum stress
S_{min}	Minimum stress
S_N	Fatigue strength at N cycles
S_r	Range of stress
S_s (or τ)	Nominal shear stress
S_t	Nominal tensile stress
S_{ty}	Tensile yield strength
S_u	Ultimate tensile strength
σ	Standard deviation, Stress
$\hat{\sigma}$	Estimate of standard deviation
σ^2	Variance
σ_x	Standard deviation of x
σ_x^2	Variance of x
T	Torque, Temperature
t	Time
τ (tau) or S_s	Shear stress
ν (nu)	Poisson's ratio

REPORT OF COMMITTEE E-11
ON
QUALITY CONTROL OF MATERIALS*

Committee E-11 on Quality Control of Materials held two meetings during the year: on November 18, 1958, and on March 25, 1959, both at ASTM Headquarters, Philadelphia, Pa.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee E-11 presented to the Society through the Administrative Committee on Standards a proposed Tentative Recommended Practice for the Acceptance of Evidence Based on the Results of Probability Sampling. The tentative recommended practice was accepted by the Standards Committee on March 25, 1959, and will appear in the 1959 Supplement to the Book of Standards, Parts 3–10, bearing the designation E 141 – 59 T.

In view of the increasing use of probability sampling in industry, there is need for a rule by which to accept or to reject evidence based on samples. The data may come from tests of samples of materials or other sources pertinent to the decision to be made.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on ASTM Problems (C. A. Bicking, chairman) is giving consideration to objections that had been raised concerning the rounding off rule in Recommended Practice E 29 – 58. One possibility is that Recommended Practice E 29 be revised to include both the present method and the previous method (now covered by American Standard Z25.1–1940), with appropriate notes to cover situations where one or the other procedure should be used.

Subcommittee II on Bulk Sampling (Louis Tanner, chairman).—This subcommittee, which is in course of organization, will establish as a permanent activity work formerly carried on in several task groups of the committee.

Respectfully submitted on behalf of the committee,

S. COLLIER,
Chairman.

J. H. DAVIDSON,
Secretary.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

REPORT OF COMMITTEE E-12
ON
APPEARANCE*

Committee E-12 on Appearance held two meetings during the year: in Boston, Mass., on June 26, 1958, and in New York, N. Y., on April 3, 1959.

Of major importance was the change in organization and activities approved at the June, 1958, meeting at Boston. The following subcommittees were set up for activities of long-range interest:

Subcommittee I on Intercommittee Relations,

Subcommittee II on Public Relations, and

Subcommittee III on Pictorial Representation.

Task groups are appointed for shorter-term projects which go out of existence upon attainment of their more limited objectives. Four task groups on specific problems have been organized as follows:

1. Manual on Appearance,
2. Color of Petroleum and Coal-Tar Resins,
3. Goniophotometric Method, and
4. Standards for Aluminum Finishes (Joint with Committee B-7 on Light Metals and Alloys, Cast and Wrought).

A draft of a proposed recommended practice of goniophotometry of reflecting objects of materials has been made available by Task Group 3. Task Group 4 has embarked upon a major research project to develop standards for aluminum finishes by utilizing and refining present methods for evaluating several aspects of appearance. The scope of this project is quite broad; an analysis of current status, evident problems, and suggested routes for their solution will be outlined for review in an early issue of the ASTM BULLETIN.[1]

STANDARD CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Standard Method of Test for 45-deg, 0-deg Directional Reflectance of Opaque Specimens by Filter Photometry (E 97 – 55).[2]

This report has been submitted to letter ballot of the committee, which consists of 75 members; 34 members returned the ballots, of whom 30 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. W. INGLE,
Chairman.

RICHARD S. HUNTER,
Secretary.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] See "Measurement of Appearance of Aluminum Surfaces," ASTM BULLETIN No. 242, December, 1959.

[2] 1958 Book of ASTM Standards, Parts 4, 5, 6, 8, and 10.

REPORT OF COMMITTEE E-13
ON
ABSORPTION SPECTROSCOPY*

Committee E-13 on Absorption Spectroscopy and most of its subcommittees met twice during the year: in New York City on November 5 to 7, 1958, jointly with the Society for Applied Spectroscopy (SAS), and on March 1 and 2, 1959, with the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. The program in New York included presentation of the Annual Award of the Society for Applied Spectroscopy to W. R. Brode, Past-Chairman of Committee E-13, now scientific advisor to the U. S. Department of State. It also included an exhibit of spectroscopic instruments, arranged by SAS and two symposia, one on Molecular Fluorescence,[1] the other on Nuclear Magnetic Resonance (NMR) Spectroscopy,[1] arranged by Subcommittees VI and VII of Committee E-13.

Organization of Subcommittee VII on Nuclear Magnetic Resonance Spectroscopy (N. F. Chamberlain, chairman) in 1958 consummated plans of Committee E-13 to serve this new and important field of spectroscopic science.

The field of nuclear magnetic resonance spectroscopy will be one of the features of a symposium on unsolved problems of spectroscopy[2] sponsored jointly by Committee E-2 on Emission Spectroscopy and Committee E-13 at the Third Pacific Area National Meeting of the Society, October 11 to 16, 1959, in San Francisco. Committee members in the western states are arranging the program. Committee E-13 plans to meet in New York City on November 5 and 6, 1959, and in Pittsburgh, Pa., on March 1, 1960.

PROPOSED PRACTICES TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only, the following proposed practices:

Proposed Recommended Practices for:

Form of ASTM Methods Relating to Absorption Spectroscopy,[3]

General Techniques of Infrared Quantitative Analysis,[4] and

General Techniques of Ultraviolet Quantitative Analysis.[5]

REVISIONS OF TENTATIVE

The committee recommends revisions as appended hereto[6] of the Tentative Definitions of Terms and Symbols Relating to Absorption Spectroscopy (E 131 – 57 T)[7] and continuation of the definitions as tentative.

---

* Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Copies of the papers are not available except possibly by correspondence with individual authors. Abstracts appear in the minutes of the Committee E-13 meeting held in New York City, Nov. 6 to 7, 1958.

[2] To be issued as separate publication *ASTM STP No. 269.*

[3] This recommended practice has not been appended to this report. Copies are available at ASTM Headquarters.

[4] See p. 610.

[5] See p. 622.

[6] The revised tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 7.

[7] 1958 Book of ASTM Standards, Part 7.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[8]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus Specifications (T. R. Harkins, chairman) is proceeding on a laboratory program with at least twenty cooperating organizations to test the practicality of the Proposed Methods for Evaluation of Spectrophotometers.[9] A separate task group will prepare an addendum on methods of test for photometric repeatability, linearity, and accuracy of infrared spectrophotometers. Plans call for submittal of the methods for publication as tentative in 1960.

Subcommittee II on Methods (R. T. O'Connor, chairman) prepared the three proposed recommendations for publication as information referred to earlier in this report. The practices for infrared and for ultraviolet spectrophotometric analyses resulted from the efforts of sections of the subcommittee, Norman Wright and R. C. Hirt, chairmen, respectively. Another section reported progress on a proposed practice with the tentative title "Identification of Materials by the Kuentzel Absorption-Spectroscopic Punched-Card Index Method."[10]

Subcommittee III on Standard Data (M. V. Otis, chairman) with the aid of volunteer workers coded over 5000 spectra during 1958. The spectra include 700 in the near-infrared region contributed by a new section of the subcommittee (Raymond Isaac, chairman) established specifically to cover this spectral region. The spectra coded in 1958 will substantially supplement the 50,000 cards now in the Wyandotte-ASTM Punched-Card Index of Absorption Spectral Data.[10]

A survey by mail of subscribers to the index indicated general satisfaction with the present volunteer system of coding and served to recruit additional volunteers. Other activities of the subcommittee include advising the Society on administration of card punching, developing a system of distributing spectra that appear in literature available only with difficulty, and investigating new methods of coding, information storage, and data retrieval.

Subcommittee IV on Nomenclature (W. P. Tyler, chairman) prepared the new items for the Tentative Definitions of Terms and Symbols Relating to Absorption Spectroscopy (E 131) as appended hereto.[6] Other terms and symbols in recommended practices published as information have priority in future deliberations.

Subcommittee V on Publications (M. G. Mellon, chairman) furnished editorial advice on the proposed practices.

Subcommittee VI on Fluorescence Spectroscopy (R. L. Bowman, chairman) arranged the earlier-mentioned symposium comprising five papers by leaders in representative segments of the subject field. The subcommittee is currently considering problems of a standard fluorescence source, suitable solvents, recommended practices, terms and symbols, and spectral indexes. Plans are being made for another symposium to be held during the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy in March, 1960.

Subcommittee VII on Nuclear Magnetic Resonance Spectroscopy (N. F. Chamberlain, chairman) was organized in November, 1958, after an inspiring symposium that included five papers by experts in the field. Task groups of the subcommittee are dealing with referencing and presentation of spectra and with nomenclature. The subcommittee plans to sponsor additional symposia in its field.

---

[8] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[9] *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 472 (1958).

[10] The Card Index may be purchased from ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 75 members; 61 members returned their ballots, of whom 55 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. J. ROSENBAUM,
Chairman.

R. F. ROBEY,
Secretary.

PROPOSED RECOMMENDED PRACTICES FOR GENERAL TECHNIQUES OF INFRARED QUANTITATIVE ANALYSIS[1, 2]

These are proposed recommended practices and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. These recommended practices are intended to provide general information on the various techniques most often used in infrared quantitative analysis. The purpose is to render unnecessary the repetition of these descriptions of techniques in individual methods for infrared quantitative analysis.

Definitions and Symbols

2. For definitions of terms and symbols, refer to the Definitions of Terms and Symbols Relating to Absorption Spectroscopy (ASTM Designation: E 131).[3]

Theory

3. Quantitative spectrophotometry is based on Beer's law which may be expressed in the following form when applied to a mixture of n components:

$$A_\lambda = a_1bc_1 + a_2bc_2 + \cdots a_nbc_n \ldots\ldots (1)$$

where:

A_λ = absorbance (total) of the sample with the spectrometer set at the wavelength λ. Absorbance, A, is defined as $A = \log_{10} \dfrac{P_o}{P}$, where P_o = incident radiant power, and P = transmitted radiant power.

a_i = absorptivity of the component i at the wavelength, λ. Absorptivity is defined as $a = \dfrac{A}{bc}$, where b = internal cell length and c = concentration.

c_i = concentration of the component i.

For best accuracy, an analytical wavelength, λ_1, is chosen where component 1 has highest absorptivity in proportion to the combined absorptivities of the other components. Similarly a wavelength λ_2 is chosen for component 2, etc. Rewriting Eq 1 for each of these n wavelengths yields a set of n equations:

$$\left.\begin{array}{r} A_1 = a_{11}b_1c_1 + a_{12}b_1c_2 + \cdots + a_{1n}b_1c_n \\ \text{(at wavelength 1)} \\ A_2 = a_{21}b_2c_1 + a_{22}b_2c_2 + \cdots + a_{2n}b_2c_n \\ \text{(at wavelength 2)} \\ \cdots\cdots\cdots\cdots\cdots\cdots \quad \cdots\cdots \\ \cdots\cdots\cdots\cdots\cdots \\ A_n = a_{n1}b_nc_1 + a_{n2}b_nc_2 + \cdots + a_{nn}b_nc_n \\ \text{(at wavelength } n\text{)} \end{array}\right\} \ldots\ldots (2)$$

In these equations the values of A_i are known (by measurement of the sample with the spectrometer), the values of a_{ij} (absorptivity at wavelength i of component j) are known from measurement of the pure individual components (see Section 6 on analytical curve preparation), and the values of b_i are the known cell lengths. The n equations of Eqs 2 can therefore be solved for the n unknown concentrations, c_j.

Since cells of fixed length are customarily used, it is usually not necessary to measure the cell lengths explicitly. The absorptivity - cell length product, $a_{ij}b_i$, may be treated as one coefficient and could be replaced in Eqs 2 by the term α_{ij}. This combined coefficient is determined from measurements of the pure individual components in the analytical cell as described in Section 6.

Apparatus

4. Reference should be made to the Proposed Methods for Evaluation of Spectro-

---

[1] These proposed recommended practices are under the jurisdiction of the ASTM Committee E-13 on Absorption Spectroscopy.

[2] Published as information, June, 1959.

[3] 1958 Book of ASTM Standards, Part 7.

photometers.[4] However, the technique descriptions given here assume only that the equipment is of the usual commercial quality.

Absorbance Measurement Methods

5. (*a*) *Point Method* **(1,2,3)**[5]:

(*1*) Set the instrument at the analytical wavelength, and with the sample cell and sample in the beam read the transmitted radiant power, P_1.

(*2*) With the same wavelength setting (and with all other instrument settings the same, for example, slit widths) read transmitted radiant power P_2 according to one of the following conditions:

If the sample is in vapor form, remove the sample by pumping and with the empty cell in the beam read radiant power, P_2.

If the sample is in solution, replace the sample solution with pure solvent in the same cell and read P_2.

If the sample is in liquid form, an alternative procedure is the following: Replace the sample and cell with a single plate of the same window material as used in the cell and read P_2. A correction constant may be required.

(*3*) Taking the above readings usually requires the subtraction of a zero reading from the transmitted power readings. This is obtained by placing an opaque shutter in the beam, which yields the reading P_3. In cases where scattered short wavelength radiation is present (spectral impurity) a radiation shutter other than metal, such as lithium fluoride (LiF), calcium fluoride (CaF_2), or barium fluoride (BaF_2) is used. This should be opaque to the radiation used but transparent to the short wavelength scattered radiation.

Transmitted radiant power, $P = P_1 - P_3$

Incident radiant power, $P_o = P_2 - P_3$

$$\text{Absorbance, } A = \log_{10} \frac{P_o}{P}$$

NOTE 1: *Scanning.*—It is obvious that the absorbance can be measured from scanned records where the same spectral interval is recorded under conditions of steps (*1*), (*2*), and (*3*).

NOTE 2: *Direct Reading of Absorbance.*—On some instruments an electrical network is provided that permits the absorbance to be read directly on a calibrated dial. In this case Steps (*2*) and (*3*) are accomplished by setting the absorbance dial to read zero and adjusting the balancing dial until the indicator (pen or galvanometer) reads zero. (The indicator is independently adjusted to zero as in Step (*3*) with a radiation shutter in the beam and the absorbance dial set at infinity. Step (*1*) is then carried out and the reading obtained by adjusting the absorbance dial until the indicator again reads null. The absorbance of the sample is then read directly from the dial. Specific instructions for commercial instruments are available in manuals provided by the manufacturers.

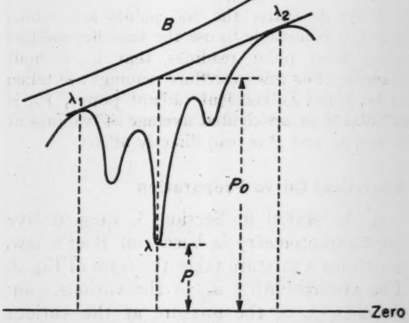


FIG. 1.—Illustration of the Base-Line Method for Absorbance Measurements.

(*b*) *Base-Line Method.*—This method **(4,5, 6)** ordinarily applies to scanned or recorded spectra, and it applies to spectra recorded by either single- or double-beam spectrometers. It consists of the following steps:

(*1*) Record an interval of the spectrum containing the band to be measured. For the initial establishing of the end points of the base line it is useful to rescan the interval with the sample removed from the beam (curve *B* of Fig. 1). It will be assumed that the radiation zero is known.

(*2*) Select two points, λ_1 and λ_2 (Fig. 1), at each side of the band to be measured, at which points the transmitted radiant power is as near as possible to curve *B* and at which the tangents to the spectrum curve parallel the curve *B*. These points, λ_1 and λ_2, should be as free as possible of absorption by all components of the sample. Draw a straight line between the two selected points. This should be tangent to the spectrum curve at the two points and should be roughly parallel to the curve *B*.

---

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 472 (1958).

[5] The boldface numbers in parentheses refer to the references listed at the end of the method.

(*3*) The values of P_o and P are obtained as indicated in the figure, and the absorbance is again given by $A = \log_{10} \frac{P_o}{P}$. The same end points λ_1 and λ_2 of the base line are, of course, used on all subsequent absorbance measurements.

Note 3.—After the end points are established, it is possible to use the base-line method using fixed point readings, that is, without scanning. For this procedure readings are taken at λ_1, λ and λ_2. Incident radiant power, P_o, is calculated as a weighted average of readings at λ_1 and λ_2, and P is read directly at λ.

Analytical Curve Preparation

6. As stated in Section 3, quantitative spectrophotometry is based on Beer's law, which for a mixture takes the form of Eqs 2. The absorptivities, a_{ij} of the various components, j, of the mixture at the various wavelengths, i, selected for the analysis must be determined from absorbance measurements made on each component taken individually. These must be measured under conditions (cell thickness, pressure, solvent, etc.) identical with those which will apply when the unknown is measured. Absorbance measurements should be made with various concentrations of the component in the cell bracketing the amounts expected in the unknown samples to be analyzed. For most accurate analysis the amount of the component in the cell in an average sample should be such as to produce absorbance near 0.434 (36.8 per cent transmitted). It is customary to plot the absorbance values of a component as ordinates versus the quantity of the component in the cell as abscissa, and this is the so-called Beer's law plot or analytical curve. The quantity of the component is usually expressed in terms of partial pressure or mole fraction, if a gas, and as concentration in grams per liter or as a weight fraction if liquid or solid. The plot thus obtained is, to a first approximation, a straight line, passing through the origin, the slope of which is the value of the product $a_{ij}b_i$ needed for the solution of Eqs 2. Treatment of the case where the plot has appreciable curvature is described in Section 7 (*d*).

(*a*) *Gases.*—Gas samples may be introduced into the cell for measurement in two different ways: (*1*) The cell is evacuated and the sample is simply admitted until a fixed standard pressure is reached; or (*2*) The sample is admitted until the pressure is some fixed fraction (for example, 20 per cent) of the total standard pressure to be used, and a nonabsorbing diluent gas, such as nitrogen, is then introduced until the total standard pressure is reached.

Analytical curves are prepared for method (*1*) by introducing a component of the mixture (in the pure state) into the cell at measured pressures and measuring the absorbances at the wavelengths selected for the analysis. These are then plotted against the pressures (which may be expressed as mole fraction) to yield the analytical curves for this component. The pressure as read by a manometer must be corrected for gas law imperfection and for temperature differences in order to be accurately proportional to the number of molecules in the absorption cell. Gas law imperfection requires that the pressure be corrected upward by a factor characteristic of the molecule. Temperature corrections are made using the ideal gas law. Analytical curves thus prepared do not take into account the possibility of band-broadening effects being different for different components. When such effects are appreciable the dilution method (*2*) is preferred.

In method (*2*), analytical curves are prepared by introducing one of the components into the cell at known pressures (up to the fixed maximum fraction, for example 20 per cent, of the total standard pressure), bringing the total pressure to the required standard value by adding the diluent gas, and measuring the absorbances at the wavelengths selected for the analysis. The absorbances are plotted against the partial pressures (or mole fractions) to produce the analytical curves.

A procedure for making standard gas mixtures, using the apparatus shown in Fig. 2, is given by the following example describing the preparation of a blend of 10 per cent methane, 5 per cent ethane, 10 per cent propane, and 75 per cent nitrogen.

(*1*) Open valve *A* and with *B* and *C* open evacuate the system and the inlet sample line to the reference gas container until the manometer indicates zero pressure.

(*2*) Close valve *A* and introduce methane into the system to a pressure of 76 mm Hg.

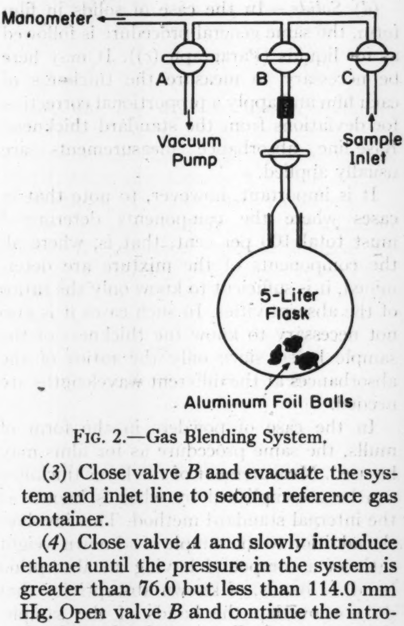


FIG. 2.—Gas Blending System.

(*3*) Close valve *B* and evacuate the system and inlet line to second reference gas container.

(*4*) Close valve *A* and slowly introduce ethane until the pressure in the system is greater than 76.0 but less than 114.0 mm Hg. Open valve *B* and continue the introduction of ethane until the pressure is 114.0 mm Hg.

(*5*) Close valve *B* and evacuate the system and inlet line.

(*6*) Close valve *A* and slowly introduce propane until the pressure is greater than 114.0 but less than 190.0 mm Hg. Open valve *B* and continue the introduction of propane until the pressure is 190.0 mm Hg.

(*7*) Close valve *B* and evacuate the system and sample inlet line.

(*8*) Close valve *A* and slowly introduce nitrogen until the pressure is greater than 190.0 but less than 760.0 mm Hg. Open valve *B* and continue the introduction of nitrogen until the pressure is 760.0 mm Hg.

(*9*) Close valve *B* and mix the sample in the blending flask thoroughly by swirling the aluminum balls.

The calculation of the mole concentrations of the gases in the synthetic blend is possible using the following equation:

$$\text{Mole per cent} = \frac{P_i}{P_t} \times 100$$

where:

P_i = pressure of individual gas in millimeters of mercury, and

P_t = total pressure of gases in millimeters of mercury.

For the example:

$$\text{Ethane, per cent} = \frac{114.0 - 76.0}{760.0} \times 100$$

$$\text{Ethane} = 5.0 \text{ mole per cent}$$

NOTE 4.—The pressure of the gas in the system must be greater than the pressure in the blending flask when valve *B* is opened to prevent back-diffusion of the gases.

NOTE 5.—It is necessary to thoroughly mix the gases in the blend by swirling the aluminum balls if an immediate analysis is to be made. Natural diffusion is relatively slow.

(*b*) *Solutions*.—One of the components (in the pure state) is introduced into the cell at a known concentration in a nonabsorbing solvent (preferably carbon disulfide (CS_2) or carbon tetrachloride (CCl_4)). The absorbance at the wavelength of the selected band is then read and plotted against the concentration. Several such points are plotted covering the range of concentrations expected in the samples to be analyzed. This constitutes the analytical curve, the slope of which is the value of the absorptivity - cell length product for this component and wavelength. Similarly, analytical plots are constructed for this component for each of the other wavelengths to be used in the analysis of the given mixture. Thus there are n plots for each component, or a total of n^2 analytical curves to be constructed, each yielding one of the values of $a_{ij}b_i$.

(c) *Liquids.*—In cases where a liquid mixture is to be analyzed without use of a diluting solvent, a complication arises because of intermolecular forces. An absorption band of a component may undergo intensity or wavelength changes, or both, when in solution in other components. It is seldom possible, therefore, to calculate accurately the absorbance contribution of a component in a mixture from its absorbance measured in the pure state. It is hence necessary to determine the absorptivities from known mixtures having proportions near those of the samples to be analyzed. The following procedure is probably the most straightforward.

Standard mixtures are prepared having concentrations of the various components covering the range expected in the unknown samples to be analyzed. Absorbances are measured at each of the wavelengths chosen for the analysis and are substituted (along with the known concentrations) into the Beer's law equations (Eqs 2), which may then be solved for the absorptivity - cell length products, $a_{ij}b_i$. The number of standard mixtures required is equal to the number, n, of components to be determined. There will thus be for each analytical wavelength a set of n equations in n unknowns. There will be n sets of these equations (since there are n wavelengths), but each set can be solved individually using the methods detailed in Section 7.

Because of the previously mentioned intermolecular influences, it is recommended that the calculation of the absorptivity - cell length products, $a_{ij}b_i$, be repeated using a second set of n standard mixtures if the concentrations in the unknowns have a wide range. The differences in values of the absorptivities thus determined will indicate the extent of intermolecular influences. In many cases it may not be possible to analyze mixtures throughout all possible concentration ranges of the components using a single set of absorptivities. Since in this procedure the absorptivity - cell length products are calculated directly, it is not necessary to plot analytical curves.

At the expense of preparing a larger number of standard mixtures, an alternative to the above procedure is to prepare standard mixtures containing only two of the components at a time. This reduces the problem to a series of sets of two simultaneous equations for two unknowns. However, all possible combinations are required in order to evaluate the intermolecular effects, and it is obviously not possible in a two-component standard mixture to have both concentrations near those of the average sample to be analyzed.

(d) *Solids.*—In the case of solids in film form, the same general procedure is followed as for liquids (Paragraph (c)). It may here be necessary to measure the thickness of each film and apply a proportional correction for deviations from the standard thickness. Base-line absorbance measurements are usually applied.

It is important, however, to note that in cases where the components determined must total 100 per cent, that is, where all the components of the mixture are determined, it is sufficient to know only the ratios of the absorptivities. In such cases it is also not necessary to know the thickness of the sample layer, since only the ratios of the absorbances at the different wavelengths are needed.

In the case of powders in the form of mulls, the same procedure as for films may be used. Measurement of the layer thickness can be accomplished by what is known as the internal standard method. This involves the addition to the sample of a known weight ratio of a compound having an absorption band, or bands, of known absorptivity that do not overlap the bands of the sample. Again, it should be noted that in cases where the components measured must total 100 per cent it is not necessary to measure the layer thickness.

In the case of powders measured in the form of pressed plates or pellets, analytical curves can ordinarily be prepared in the same manner as for solutions (Paragraph (b)).

Calculation Methods

7. (*a*) *Matrix Inversion.*—After the absorptivity - cell length products, $a_{ij}b_i$, have been determined for a given set of mixture components, the numerical values are substituted into the Beer's law equations (Eqs 2). (The concentrations, c_j will now be considered as weight fractions and, hence, each will have a maximum value of unity.) One calculation procedure is to solve these equations for the concentrations, c_j explicitly in terms of the absorbances,[‡] A_i. This is known as matrix inversion. The solved (or inverted) equations will have the form:

$$\left.\begin{array}{l} c_1 = A_1F_{11} + A_2F_{12} + \cdots + A_nF_{1n} \\ c_2 = A_1F_{21} + A_2F_{22} + \cdots + A_nF_{2n} \\ \dots\dots\dots\dots\dots\dots\dots\dots\dots \\ c_n = A_1F_{n1} + A_2F_{n2} + \cdots + A_nF_{nn} \end{array}\right\} \quad (3)$$

where the F_{ji} will be known numbers. Thenceforth, for any individual sample, the concentrations can be calculated simply by substituting the measured values of the absorbances, A_i, into these equations.

The F_{ji} can be calculated in the following manner (7), taking as example the case of a mixture containing five components. First, construct the following matrix, called the *B*-array:

$$\begin{array}{ccccccccc} \underline{B_{11}} & B_{21} & B_{31} & B_{41} & B_{51} & B_{61} & \cdots & B_{10\,1} \\ B_{12} & \underline{B_{22}} & B_{32} & B_{42} & B_{52} & B_{62} & \cdots & B_{10\,2} \\ \dots & \dots & \dots & \dots & \dots & \dots & & \dots \\ B_{15} & B_{25} & B_{35} & B_{45} & \underline{B_{55}} & B_{65} & \cdots & B_{10\,5} \end{array}$$

where the B_{uv} have the values:

$$\begin{array}{cccccccccc} \underline{\alpha_{11}} & \alpha_{21} & \alpha_{31} & \alpha_{41} & \alpha_{51} & 1 & 0 & 0 & 0 & 0 \\ \alpha_{12} & \underline{\alpha_{22}} & \alpha_{32} & \alpha_{42} & \alpha_{52} & 0 & 1 & 0 & 0 & 0 \\ \alpha_{13} & \alpha_{23} & \underline{\alpha_{33}} & \alpha_{43} & \alpha_{53} & 0 & 0 & 1 & 0 & 0 \\ \alpha_{14} & \alpha_{24} & \alpha_{34} & \underline{\alpha_{44}} & \alpha_{54} & 0 & 0 & 0 & 1 & 0 \\ \alpha_{15} & \alpha_{25} & \alpha_{35} & \alpha_{45} & \underline{\alpha_{55}} & 0 & 0 & 0 & 0 & 1 \end{array}$$

This is called the original array, the underlined terms are called the diagonal terms, the column on the left (B_{11}, B_{12}, $\cdots$ B_{15}) is called the first column, and the row on the top (B_{11}, B_{21}, $\cdots$ $B_{10\,1}$) is called the first row. The first term of a column is the top term and the first term of a row is the left-hand term.

From this original array construct, column by column, a second array, the *S*-array,

$$\begin{array}{cccccccccc} \underline{S_{11}} & S_{21} & S_{31} & S_{41} & S_{51} & S_{61} & 0 & 0 & 0 & 0 \\ S_{12} & \underline{S_{22}} & S_{32} & S_{42} & S_{52} & S_{62} & S_{72} & 0 & 0 & 0 \\ S_{13} & S_{23} & \underline{S_{33}} & S_{43} & S_{53} & S_{63} & S_{73} & S_{83} & 0 & 0 \\ S_{14} & S_{24} & S_{34} & \underline{S_{44}} & S_{54} & S_{64} & S_{74} & S_{84} & S_{94} & 0 \\ S_{15} & S_{25} & S_{35} & S_{45} & \underline{S_{55}} & S_{65} & S_{75} & S_{85} & S_{95} & S_{105} \end{array}$$

in the following manner:

(*1*) The first column of the second array is the same as the first column of the original array. For example,

$$S_{15} = B_{15}$$

(*2*) Each term (except the first) in the first row of the second array is equal to the corresponding term of the original array divided by the first term in this row. For example,

$$S_{41} = \frac{B_{41}}{S_{11}}$$

(*3*) Each diagonal term in the second array is equal to the corresponding term in the original array minus the product of the first term in the same row and the first term in the same column of the second array, minus the product of the second term in the same row and the second term in the same column, etc, until no more such products are possible. For example,

$$S_{55} = B_{55} - (S_{15})(S_{51}) - (S_{25})(S_{52}) - (S_{35})(S_{53}) - (S_{45})(S_{54})$$

(*4*) Each term below the diagonal is calculated in the same manner as the diagonal terms. For example,

$$S_{45} = B_{45} - (S_{15})(S_{41}) - (S_{25})(S_{42}) - (S_{35})(S_{43})$$

(*5*) Each term in the second array above and to the right of the diagonal is obtained in the same manner as the diagonal terms, except that the number so obtained is divided by the diagonal term in the same row as the term being computed. For example,

$$S_{84} = \frac{0 - (S_{14})(0) - (S_{24})(0) - (S_{34})(S_{83})}{S_{44}}$$

and

$$S_{65} = B_{65}$$
$$- \frac{(S_{15})(S_{61}) - (S_{25})(S_{62}) - (S_{35})(S_{63}) - (S_{45})(S_{64})}{S_{55}}$$

Construct a final array, the F-array,

$$\begin{array}{ccccc} F_{11} & F_{21} & F_{31} & F_{41} & F_{51} \\ F_{12} & F_{22} & F_{32} & F_{42} & F_{52} \\ F_{13} & F_{23} & F_{33} & F_{43} & F_{53} \\ F_{14} & F_{24} & F_{34} & F_{44} & F_{54} \\ F_{15} & F_{25} & F_{35} & F_{45} & F_{55} \end{array}$$

according to the following procedure:

(*1*) The last row in the F (final) array is the same as the last five terms of the last row in the S-array. For example, $F_{45} = S_{95}$. This correspondence of columns holds throughout the remaining computation, that is, column five in the F-array corresponds to column ten in the S-array, column two corresponds to column seven, etc.

(*2*) Compute all columns of the F-array from the bottom up. Each term (except the bottom row) in the F-array is equal to the corresponding term in the second array minus the product of the first term to the right of the diagonal term in the same row in the S-array and the first term in the F-array below the term being determined, minus the product of the second term to the right of the diagonal term in the S-array and the second term in the F-array below the term being determined, etc., until no more such products are possible. For example,

$$F_{33} = S_{83} - (S_{43})(F_{34}) - (S_{53})(F_{35})$$

and

$$F_{52} = 0 - (S_{32})(F_{53}) - (S_{42})(F_{54}) - (S_{52})(F_{55})$$

The second array can be checked after the completion of each row. The product of the first term in the row (second half) of the S-array to be checked and the first term in the corresponding column of the B-array (first half) is added algebraically to the product of the second term in the row and the second term in the column, etc. The sum of these products should equal 1.000. For example,

$$(S_{64})(B_{41}) + (S_{74})(B_{42}) + (S_{84})(B_{43}) + (S_{94})(B_{44}) = 1.000$$

Deviations of plus or minus two units in the last decimal place to which the computations are carried are neglected.

The final array can be checked after the completion of each row. The product of the first term in the row of the F-array to be checked and the first term in the corresponding column of the B-array is added algebraically to the product of the second term in the row and the second term in the column, etc. The algebraic sum of these products should equal 1.000. For example,

$$(F_{14})(B_{41}) + (F_{24})(B_{42}) + (F_{34})(B_{43}) + (F_{44})(B_{44}) + (F_{54})(B_{45}) = 1.000$$

and

$$(F_{11})(B_{11}) + (F_{21})(B_{12}) + (F_{31})(B_{13}) + (F_{41})(B_{14}) + (F_{51})(B_{15}) = 1.000$$

The last row of the F-array need not be checked since it was taken term for term from the S-array which has already been checked.

(*b*) *Successive Approximation.*—Another calculation procedure for multicomponent mixtures involves solving the Beer's law equations (Eqs 2) for each individual unknown sample. In this case, the known values of the absorptivity - cell length products, α_{ij}, and the measured values of the absorbances, A_i (for the individual sample) are substituted into the Beer's law equations. These equations are now solved by a method of successive approximations as follows, taking as example a four-component mixture:

$$A_1 = \alpha_{11}c_1 + \alpha_{12}c_2 + \alpha_{13}c_3 + \alpha_{14}c_4 \quad \text{(at wavelength 1)} \ldots. (4)$$

$$A_2 = \alpha_{21}c_1 + \alpha_{22}c_2 + \alpha_{23}c_3 + \alpha_{24}c_4 \quad \text{(at wavelength 2)} \ldots. (5)$$

$$A_3 = \alpha_{31}c_1 + \alpha_{32}c_2 + \alpha_{33}c_3 + \alpha_{34}c_4 \quad \text{(at wavelength 3)} \ldots. (6)$$

$$A_4 = \alpha_{41}c_1 + \alpha_{42}c_2 + \alpha_{43}c_3 + \alpha_{44}c_4 \quad \text{(at wavelength 4)} \ldots. (7)$$

where all numbers are known except the c_j.

First Approximation:

$$c_1 = \frac{A_1}{\alpha_{11}}, \quad c_2 = \frac{A_2}{\alpha_{22}}, \quad c_3 = \frac{A_3}{\alpha_{33}}, \quad c_4 = \frac{A_4}{\alpha_{44}}$$

Second Approximation:

(*1*) Substitute the first approximation values of c_2, c_3 and c_4 in Eq 4 and solve for c_1. This value is the second approximation for c_1.

(*2*) Substitute into Eq 5 the second approximation value of c_1 and the first approximation values of c_3 and c_4. Solving this equation for c_2 yields the second approximation value for c_2.

(*3*) Substitute into Eq 6 the second approximation values of c_1 and c_2 and the first approximation value of c_4. Solving this equation for c_3 yields the second approximation value for c_3.

(*4*) Substitute into Eq 7 the second approximation values of c_1, c_2, and c_3. Solving for c_4 yields the second approximation value for c_4.

Third Approximation:

Substitute into Eq 4 the second approximation values of c_2, c_3, and c_4 and solve for c_1. This value is the third approximation for c_1.

Solve for the third approximations of c_2, c_3, and c_4 in the same manner as described for the second approximations.

Final Solution:

Repeat the determination of higher approximations until there is no significant change in the values.

(*c*) *Graphical.*—A successive approximation method equivalent to that described in Paragraph (*b*) can be carried out graphically in cases where analytical curves have been prepared. In such cases there will be a set of *n* (for *n* components) Beer's law plots drawn for each of the *n* wavelengths chosen for the analysis. In each of these sets of plots one will usually have a considerably greater slope than the others, since this represents the greater absorptivity of one component at the wavelength of the absorption band chosen for its analysis. This will be referred to as the primary plot for the given component, while the plots for other components at this wavelength will be referred to as secondary plots.

Again, taking as example a four-component case, assume that absorbances A_1, A_2, A_3, and A_4 have been measured at the respective wavelengths chosen for the analysis.

First Approximation:

Read the values of the concentrations from the various primary plots using the measured absorbances. These are the first approximations.

Second Approximations:

At the primary wavelength of component 1, read the absorbances of components 2, 3 and 4, using the first approximation values of the concentrations of these components. Subtract the sum of these absorbances from A_1 and read the value of c_1, using this corrected absorbance. This is the second approximation for c_1.

At the primary wavelength of component 2, read the absorbances of components 1 (using second approximation for c_1), 3, and 4 (using the first approximations for c_3 and c_4). Subtract the sum of these absorbances from A_2 and read the value of c_2 using this corrected absorbance. This is the second approximation for c_2.

Repeat these operations for the other second approximations.

Final Solution:

Repeat the above operations for the third and higher approximations until there is no significant change in the values.

(*d*) *Correction for Curvature of Beer's Law Plots.*—In some cases a Beer's law plot of one or more components of a mixture will exhibit curvature to such an extent that the value of the slope (absorptivity - cell length product) may differ appreciably between low and high concentrations. If the graphical successive approximation method (Paragraph (*c*)) is used, no change in the procedure is required. If the concentrations of such a component in the samples to be analyzed usually fall in the low range, it is permissible to use the value of the slope for this low range without any correction to the above methods of calculation (Paragraphs (*a*) and (*b*)). If, however, the concentration is larger, it is necessary to apply a correction, and the following method is recommended:

Assume that the concentration of the component under consideration ranges in the samples to be analyzed between c_1 and c_2, Fig. 3. Draw a straight line between A_1 and A_2 (or the straight line approximating the curve between A_1 and A_2 most closely). The slope of this line is the absorptivity - cell length product that is to be used in the Beer's law equations. The intercept of this line with the absorbance axis yields the value, A_0, of a correction term which must be subtracted from the measured absorbance of the sample at the analytical wavelength of the component, that is, $A - A_0$ is substituted for A in the Beer's law equation for this analytical wavelength. If the concentration of the component under consideration should happen to fall considerably outside the range c_1 to c_2 (in a sample analyzed) it will be necessary to repeat the above procedure of finding the slope and intercept for the new concentration range.

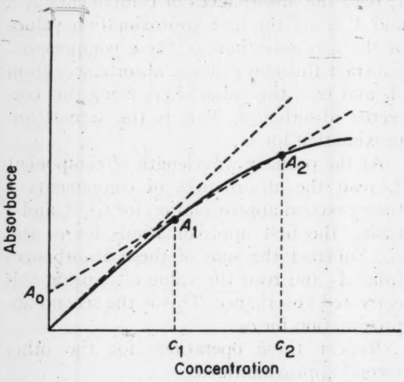


FIG. 3.—Plot of Absorbance *Versus* Concentration.

Another method of correction for curvature, which applies over the entire concentration range (with varying degrees of accuracy), requires determining an equation of the type, $A = A_m + kA_m^2$, that best approximates the Beer's law plot (8). Here, A_m is the measured absorbance, k is a constant determined from the Beer's law plot, and A is the value of absorbance to be used in the Beer's law equations.

Special Techniques

8. (*a*) *Reference Standard Method.*—This method can be regarded as a special case of the point method of absorbance measurement in which a known mixture or reference standard material is used in Step (*2*) of Section 5 (*a*) instead of pure solvent, empty cell, or dummy cell. The standard material thus used for reference should be as nearly identical to the average composition of the samples being measured as possible. The reference standard material is usually measured in the same cell as the sample.

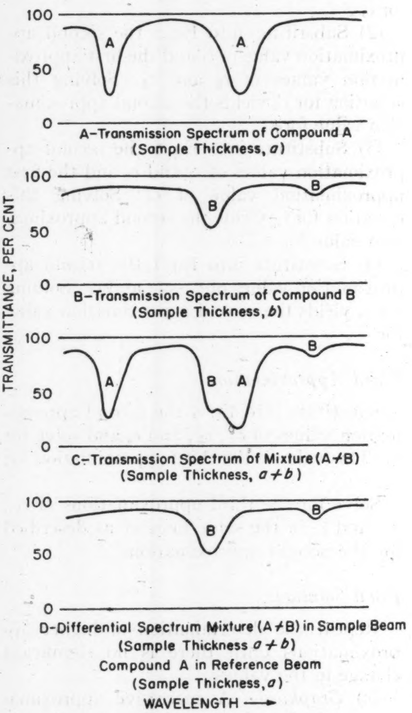


FIG. 4.—Illustration of Measurements by Differential Method.

The value of log P_o/P thus obtained is the *difference* between absorbance of sample and absorbance of reference standard. If the absorbance of sample is less than that of the reference standard log P_0/P will be negative. Since the absorbance of the reference standard is a known quantity it is necessary only to add to it (algebraically) the above measured difference in order to obtain the absorbance of the sample.

NOTE 6.—This method is usually expected to yield high accuracy since it allows a nearly null comparison of sample and standard and does not require intervening measurement of the total incident radiant power.

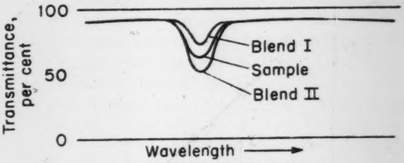
NOTE 7.—The base-line method can also be applied in the reference standard method, in which case the absorbances of sample and standard are both obtained in quick succession. Here the nearly null comparison of sample and standard may also be expected to yield higher accuracy of absorbance measurement.

(*b*) *Differential Method.*—In the differential method, the difference in the radiant power transmitted by a sample and a reference cell is measured directly by means of a double-beam spectrometer. This is illustrated in Fig. 4 for an ideal mixture which does not show solvent effects (**9,10**).

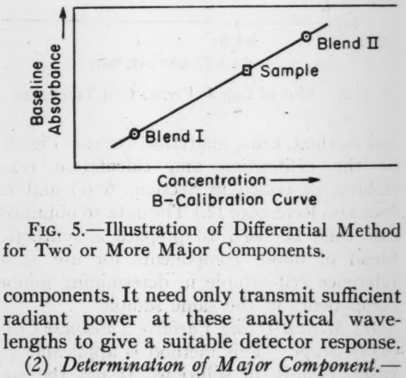
Since the composition and absorbance of the material in the reference cell is known, the differential spectrum can be used to obtain the composition of the sample. The procedure may be identical with that of the reference standard method (Paragraph (*a*)), except that thicker cells are usually employed in order to maximize the differential absorbance measured. However, the method is employed most often for partial rather than complete analyses. In these cases one of two principal procedures is employed, depending upon whether major or minor components are to be determined (**11,12**).

(*1*) *Determination of Minor Components.*—In this application, the reference cell is filled with the pure major component. The cell length is chosen so that the amount of major component in the reference beam is the same as that in the sample beam. This may be accomplished most easily by using a variable path-length reference cell and adjusting its thickness so that the strongest bands of the major component are removed from the spectrum. The resulting spectrum of the minor components is then measured and interpreted quantitatively by the usual procedures. Analytical curves are prepared according to the procedure outlined in Section 6 (*b*) for solutions, regarding the major component as the solvent. The base-line method described in Section 5 (*b*) is usually used for absorbance measurements.

It is obvious that this method can be applied to solutions. The advantage of the differential technique is that the solvent or major component need not be transparent at the analytical wavelengths of the other components. It need only transmit sufficient radiant power at these analytical wavelengths to give a suitable detector response.



A-Differential Spectra



B-Calibration Curve

FIG. 5.—Illustration of Differential Method for Two or More Major Components.

(*2*) *Determination of Major Component.*—In this application, both reference and sample cells contain the component to be determined. The method consists essentially of measuring the difference in absorbance at the analytical wavelength between the sample and a reference standard. The concentration is then determined from a calibration curve prepared from similar mixtures of known concentration (Fig. 5). If interference from minor components at the analytical wavelength is small, the reference cell need only contain the major component; however, for maximum accuracy the minor constituents should also be present in the reference sample in approximately the same concentrations as occur in the sample being analyzed. The sample cell thickness is chosen to give a sample absorbance in the range of 1.0 to 1.4 at the analytical wavelength, (higher absorbances give greater analytical sensitivity, provided adequate detector response is obtained). The reference cell thickness is usually adjusted to give differential absorbance values in the range of 0.01 to 0.10.

Samples having two or more major components can also be analyzed by the differential method, using analytical curves (Fig. 5) or the calibration and calculation procedures described in Sections 6 (*c*) and 7. (See also Reference 12.) The data so obtained can then be used to prepare a synthetic blend of major components for use as a reference cell sample in determining minor components in the same sample.

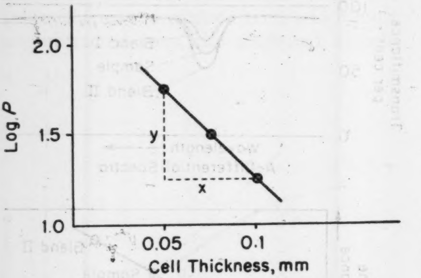


FIG. 6.—Plot of Log *P Versus* Cell Thickness.

(*c*) *Method Using Variable Thickness Cell:*

(*1*) *Scope.*—This method is applicable to gases, liquids, or solutions. It has the advantages that no blank or dummy cell measurements are required, and that absorption by the cell itself need not be measured (13).

(*2*) *Description and Theory of the Method.*—In this method a cell is needed which can be varied in thickness by at least a factor of 2, and which provides an accurate measurement of the difference in thickness between different settings. Absolute thickness measurement is not required.

The method consists of plotting log *P* at the wavelength of the selected band *versus* the thickness readings of the cell, yielding a straight line as shown in Fig. 6. The incident radiant power, P_o, is held constant during these measurements, but its value does not enter into the calculation. This follows from Beer's law which states that at any wavelength:

$$\log \frac{P_o}{P} = abc + K$$

or

$$\log P = -abc - K + \log P_o,$$

where:

K = the absorbance of the cell.

Plotting log *P* against the cell thickness therefore yields a straight line of which the negative slope (y/x, Fig. 1) is equal to ac. With a single component in the cell (or a single component in the common solvent) $c = 1$, and these plots yield the values of a_{ij}.

(*3*) *Absorbance Measurement*—With the unknown mixture in the cell, plots of log *P* versus the cell thickness reading are made at each of the *n* wavelengths chosen for the *n* components. The negative slopes of the lines plotted for each wavelength are the absorbances per unit thickness of cell and yield the following relations:

$$\begin{aligned} -\text{slope at } \lambda_1 &= c_1a_{11} + c_2a_{21} + \cdots + c_na_{n1} \\ -\text{slope at } \lambda_2 &= c_1a_{12} + c_2a_{22} + \cdots + c_na_{n2} \\ &\cdots\cdots\cdots\cdots \\ -\text{slope at } \lambda_n &= c_1a_{1n} + c_2a_{2n} + \cdots + c_na_{nn} \end{aligned}$$

Since the values of a_{ij} are known, these equations may be solved in the usual manner for the concentrations.

REFERENCES

(1) R. R. Brattain, R. S. Rasmussen, and A. M. Cravath, "A Spectrophotometric Method for the Analysis of Multicomponent Mixtures and Its Infrared Absorption," *Journal of Applied Physics*, Vol. 14, p. 418 (1943).

(2) J. R. Nielsen and D. C. Smith, "Analysis by Infrared Spectroscopy. A New Method Applied to Mixtures of Nitroparaffins," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 15, p. 609 (1943).

(3) J. W. Kent and J. Y. Beach, "New Infrared Spectrometric Quantitative Analyses of Multicomponent Liquid Hydrocarbon Mixtures," *Analytical Chemistry*, Vol. 19, p. 290 (1947).

(4) N. Wright, "Application of Infrared Spectroscopy to Industrial Research," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 13, p. 1 (1941).

(5) J. J. Heigel, M. F. Bell, and J. U. White, "Application of Infrared Spectroscopy to the Analysis of Liquid Hydrocarbons," *Analytical Chemistry*, Vol. 19, p. 293 (1947).

(6) R. A. Friedel, Lois Pierce, and J. J. McGovern, "Infrared Analysis of Phenol, Cresols, Xylenols and Ethylphenols," *Analytical Chemistry*, Vol. 22, p. 418 (1950).

(7) P. D. Crout, "A Short Method for Evaluating Determinants and Solvent Systems of Linear Equations with Real or Complex Coefficients," *Transactions*, American Institute Electrical Engineers, Vol. 60, p. 1235 (1941).

(8) J. H. Lee, "Determination of Extinction Corrections in Infrared Analysis of Gaseous Hydrocarbon Mixtures," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 18, p. 659 (1946).

(9) D. Z. Robinson, "Quantitative Analysis with Infrared Spectrophotometers. Differential Analysis," *Analytical Chemistry*, Vol. 23, p. 273 (1951).

(10) D. Z. Robinson, "Quantitative Analysis with Infrared Spectrophotometers. Differential Analysis," *Analytical Chemistry*, Vol. 24, p. 619 (1952).

(11) C. F. Hammer and H. R. Roe, "A Differential Method for Accurate Quantitative Infrared Analysis. Assay for Cyclohexane in Petroleum Concentrates," *Analytical Chemistry*, Vol. 25, p. 668 (1953).

(12) M. Beroza, "Differential Multicomponent to Spectrophotometry. Spectrophotometric Method for Determination of Benzyl Benzoate and N-Butyl Acetanilitride in Clothing Impregnant M-196°," *Analytical Chemistry*, Vol. 25, p. 112 (1953).

(13) J. R. Nielsen and D. C. Smith, "Analysis of Infrared Spectroscopy. A New Method Applied to Mixtures of Nitroparaffin," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 15, p. 609 (1943).

PROPOSED RECOMMENDED PRACTICES FOR GENERAL TECHNIQUES OF ULTRAVIOLET QUANTITATIVE ANALYSIS[1, 2]

These are proposed recommended practices and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. These recommended practices are intended to provide general information on the various techniques most often used in ultraviolet quantitative analysis. The purpose is to render unnecessary the repetition of these descriptions of techniques in individual methods for ultraviolet quantitative analysis.

Principle

2. Quantitative ultraviolet analysis is based upon the absorption law, known as Beer's law. The units of this law are defined in the Definitions of Terms and Symbols Relating to Absorption Spectroscopy (ASTM Designation: E 131).[3] Beer's law (Note), when applied to a mixture of n components, may be expressed in the following form (see Section 8):

$$A = a_1bc_1 + a_2bc_2 + \cdots + a_nbc_n$$

NOTE.—Detailed discussion of the origin and validity of Beer's law may be found in the books and articles listed in the bibliography at the end of these recommended practices.

Sample Preparation

3. Weigh by difference the specified amount of the sample (solid or liquid) into the volumetric flask. Add the solvent and shake to dissolve the sample completely. Fill to the mark with solvent. (Solvent and flask should be at approximately the same temperature as the spectrophotometer.) If needed, a dilution should be made with a calibrated pipet and volumetric flask, using adequate volumes for accuracy. Fill the absorption cell with the solution and fill the comparison or blank cell with the pure solvent, which should match that used for the solution as closely as possible.

Base-Line and Cell Checking

4. The balance of the spectrophotometer should be checked at several wavelengths without cells in the beam for manually operated instruments; a base-line scan should be made with automatic recording instruments. Prior to analytical use, the cells should be checked against each other with pure solvent in each. A check for cell length matching may be made by examining a strongly absorbing solution versus a pure solvent, and then emptying, cleaning, and reloading solvent and sample into the other cells. Absorbance readings should check within 1 per cent.

Analytical Wavelengths

5. (*a*) Analytical wavelengths are those wavelengths at which absorbance readings are taken for use in calculations. These may include readings taken for purposes of background corrections. The analytical wavelengths are frequently chosen at absorption maxima; this is not necessarily always done. For example, use of isoabsorptive or isosbestic points is frequently helpful.

(*b*) On manually operated spectrophotometers, record the absorbance readings at the specified analytical wavelengths, operating the instrument in accordance with the recommendations of the manufacturer or the Proposed Methods for Evaluation of Spec-

---

[1] These proposed recommended practices are under the jurisdiction of the ASTM Committee E-13 on Absorption Spectroscopy.

[2] Published as information, June, 1959.

[3] 1958 Book of ASTM Standards, Part 7.

trophotometers.[4] On automatic recording spectrophotometers, record the absorbance readings from the chart at the analytical wavelengths. The wavelength region scanned should be somewhat greater than that required to contain only the analytical wavelengths. Care must be taken to start the chart and scanning drives exactly together. End the scan well before the slit has opened fully.

(*c*) Absorbance values should be used only if they fall within the acceptably accurate range of the particular spectrophotometer used. Generally, this means between absorbances of 0.1 and 1.0. (Refer to the Proposed Methods for Evaluation of Spectrophotometers.[4]. If the absorbance is too low, use either a longer absorption cell or prepare a new solution of higher concentration. If the absorbance is too high, use a shorter cell or make a quantitative dilution.

Slit Widths

6. If a fixed slit width is specified in the analytical method, this should be used, and the sensitivity control properly adjusted for balance. If no slit width is specified, the value of the slit width should be recorded. (Changes in the day-to-day values of slit width are often a clue to instrumental defects, dirt accumulation on optical parts, or deposition of films on the interior of the reference absorption cells.)

Solvents and Solvent Effects

7. (*a*) The ultraviolet absorption spectrum of a compound will vary in different solvents depending on the chemical structures involved. Nonpolar solvents have the least effect on the absorption spectrum. Nonpolar molecules in most instances are not affected in polar solvents. However, polar molecules in polar solvents may show marked differences in their spectra. Any interaction between solute and solvents leads to a broadening and change in structural resolution of the absorption bands. Ionic forms may be created in acidic or basic solutions. In addition, there are possible chemical reactions between solute and solvent, and also photochemical reactions arising from either room illumination or the short wavelengths in the beam of the spectrophotometer. It is important that the solvent used be specified in recording spectral data. (The change in spectra between acidic and basic conditions may sometimes be employed in multicomponent analysis.)

(*b*) Common, commercially available solvents of "spectroscopic purity" are listed in Table I. The short wavelength limit is approximate, and refers to the wavelength at which a 1-cm light path length gives an absorbance of unity.

TABLE I.—SOLVENTS.[a]

Solvent	Cutoff, mμ
Pyridine	305
Tetrachloroethylene	290
Benzene	280
N,*N*-Dimethylformamide	270
Carbon tetrachloride	265
Methyl formate	260
Chloroform	245
Dichloromethane	235
Ethyl ether	220
Acetonitrile	215
Isopropyl alcohol	210
Ethyl alcohol	210
Methyl alcohol	210
Cyclohexane	<210
*Iso*octane	<210

[a] Procedures for special purification of solvents for further improvement in the wavelength limit are given in the literature (**11,12**). Several companies now supply solvents of spectroscopic purity.[5]

(*c*) Water, and 0.1 *N* solutions of hydrochloric acid, sulfuric acid, and sodium hydroxide are commonly used as solvents. Buffered solutions, involving nonabsorbing materials, are frequently used; both the composition of the buffer and the measured pH should be specified. Mixtures of 0.1 *N* dihydrogen sodium phosphate and 0.1 *N* hydrogen disodium phosphate are useful in

---

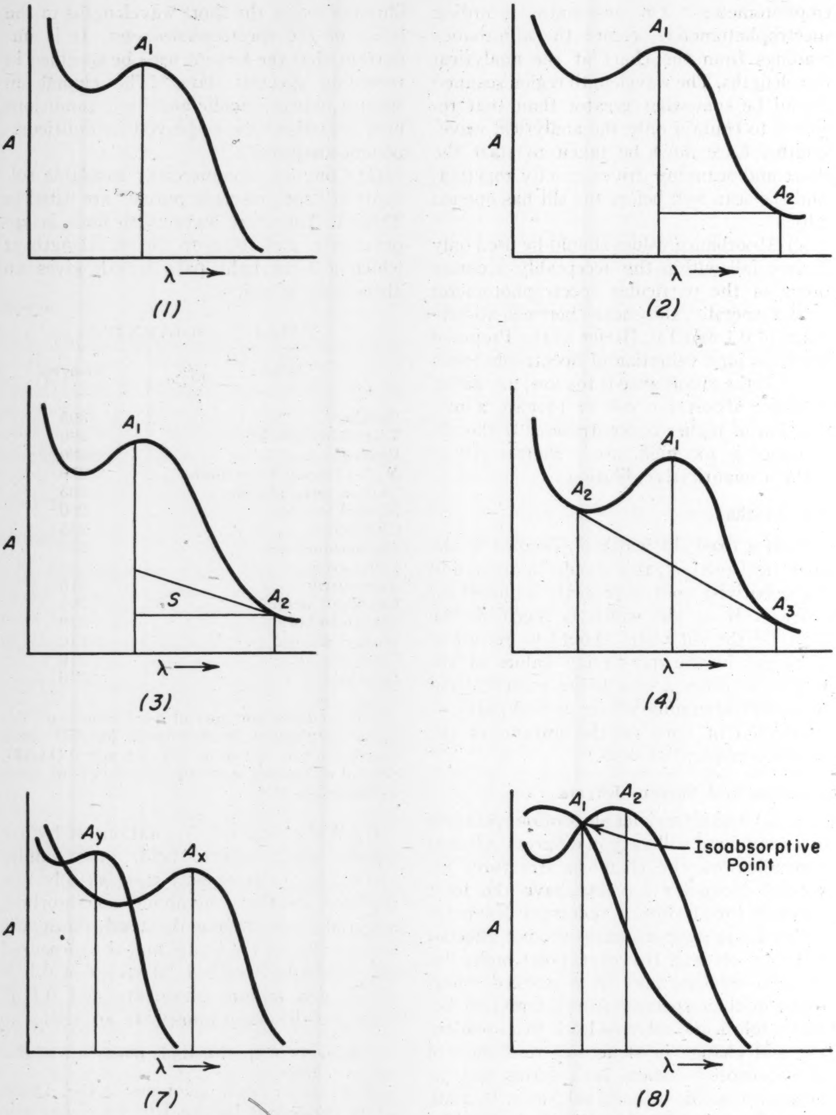
[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 472 (1958).

[5] Solvents of spectroscopic purity are available, as follows:

Solvents for Spectrophotometric Use, Distillation Products Industries, Division of Eastman Kodak Co., Rochester 3, N. Y.

Spectro Grade Solvents, Fisher Scientific Co., 717 Forbes Ave., Pittsburgh 19, Pa.

Spectroquality Solvents, Matheson, Coleman and Bell, 11-38 31st Ave., Long Island City 6, N. Y.


A
A₁
λ →
(1)
A
A₁
A₂
λ →
(2)
A
A₁
S
A₂
λ →
(3)
A
A₁
A₂
A₃
λ →
(4)
A
Aᵧ
Aₓ
λ →
(7)
A
A₁
A₂
Isoabsorptive Point
λ →
(8)

(*1*) One Component, No Background Correction.

(*2*) One Component, Simple Background Correction.

(*3*) One Component, with Slope-Type Background Correction.

(*4*) One Component, with Linear Background Correction.

(*7*) Two Components, with Overlapping Absorption for Only One Component.

(*8*) Two Components, with Mutually Overlapping Absorption.

FIG. 1—Illustrations of Types of Analysis.

the 4.5 to 8.9 pH range. A table of nonabsorbing buffers has been presented by Abbott (**8**).[6]

Calculations

8. (*a*) Quantitative analysis by ultraviolet spectrophotometry depends upon Beer's law. The terms and symbols used are those defined in the Definitions of Terms and Symbols Relating to Absorption Spectroscopy (ASTM Designation: E 131).[3] According to Beer's law:

$$A = abc = \frac{\epsilon}{M} \times bc$$

where:

A = absorbance,
a = absorptivity,
b = cell length in centimeters,
c = concentration in grams per liter,
ϵ = molar absorptivity, and
M = molecular weight.

In practice, a distinction must be made between c, the concentration of the absorbing material in the cell at the time of observation, and the concentration of the *absorbing material* in the *sample as received*. This is here designated as C. The solution to be examined has a concentration of *sample* in solution, here designated as C_s.

$$c = \frac{A}{ab}$$

$$C\text{, per cent} = \frac{c}{C_s} \times 100 = \frac{A}{abC_s} \times 100$$

(*b*) If one or more dilutions have been made, the quantity called the dilution factor must be included. Dilution factor, f, is the ratio of the final volume to the initial volume. If more than one dilution is performed, the dilution factor is the product of the factors from each dilution. If dilutions are made, the equation becomes:

$$C\text{, per cent} = \frac{c}{C_s} \times 100 = \frac{Af}{abC_s} \times 100$$

Note that c and C_s have the dimensions of grams per liter.

(*c*) *Standards.*—The absorptivity of the absorbing material, the concentration of which it is desired to determine, is obtained by examination of a pure sample of this material which is called a standard. However, if no such pure material is available, the best available sample is used, or a value of the absorptivity is taken from the literature. Care should be taken to specify this, such as reporting values as "percentage against standard" or by noting that the accuracy of the analysis is dependent upon a published value of the absorptivity or molar absorptivity. (A reference must be cited.)

(*d*) *Types of Analyses* (*see Fig. 1*):

(*1*) *One Component, No Background Correction:*

$$C\text{, per cent} = \frac{Af}{abC_s} \times 100$$

(*2*) *One Component, Simple Background Correction:*

$$C\text{, per cent} = \frac{(A_1 - A_2) \times f}{a_1 bC_s} \times 100$$

where the subscripts refer to analytical wavelengths. The term A_2 is the absorbance at the wavelength used for making a simple subtractive correction. It is usually selected from examination of the spectral curve of the standard, at a wavelength longer than that of A_1, preferably where a_2 is equal to or less than $a_1/100$.

(*3*) *One Component with Slope-Type Background Correction:*

$$C\text{, per cent} = \frac{(A_1 - SA_2) \times f}{a_1 bC_s} \times 100$$

where:

S = slope between wavelengths 1 and 2 for the background.

The background absorption is usually *not* linear between the analytical wavelength and the wavelength at which a simple subtractive background correction may be obtained. When it is possible to determine the slope between wavelengths 1 and 2 by observation of the samples that do not contain the absorbing material that is to be determined, this may be used as a correction for the background absorption.

---

[6] The boldface numbers refer to the bibliography at the end of these recommended practices.

(*4*) *One Component with Linear Background Correction:*

$$C\text{, per cent} = \frac{A_1 - \dfrac{A_2 + A_3}{2}}{abC_s} \times 100$$

This method is especially effective with materials that have sharp bands. Wavelengths 2 and 3 are selected to the long and short wavelength sides of the analytical wavelength 1, usually at minima. The absorptivity a is here the "effective" absorptivity as determined on a pure sample, using the corrections, and is somewhat lower than the true or absolute absorptivity.

(*5*) *One Component with Background Correction from Outside Data:*

$$C\text{, per cent} = \frac{(A - X) \times f}{abC_s} \times 100$$

This is a general case in which some empirical correction may be derived from data other than spectrophotometric, and is applied as an effective absorbance which is subtracted from the observed. As an example, the concentration of a known interfering material may be determined by titration, and the absorbance due to this calculated, and then subtracted.

(*6*) *Two Components, with No Overlapping Absorption.*—Apply method (*1*) twice, at the two analytical wavelengths. This is an almost impossible case, except when the relative concentrations of the two components are such that the product of absorptivity and concentration of one component at a given wavelength is more than 100 times the product for the other component, allowing the latter to be neglected.

(*7*) *Two Components, with Overlapping Absorption for Only One Component.*—Determine the component with no interference (component x) at an analytical wavelength selected to allow no contribution from component y.

$$C_x\text{, per cent} = \frac{Af}{a_1 bC_s} \times 100$$

Calculate the contribution of this component to the observed absorbance at the other analytical wavelength, where both components are absorbing, as follows:

$$A_{2x} = a_{2x}\, bc_x$$

Calculate the concentration of component y from

$$C_y\text{, per cent} = \frac{(A_2 - A_{2x}) \times f}{a_2 bC_s}$$

(*8*) *Two Components, with Mutually Overlapping Absorption.*—Use the absorbance-ratio method (graphical) described in the literature (**10**), or by simultaneous equations as follows:

$$C_x\text{, per cent} = \frac{a_{y_2} A_1 - a_{y_1} A_2}{bC_s \times (a_{y_2} a_{x_1} - a_{y_1} a_{x_2})} \times 100$$

$$C_y\text{, per cent} = \frac{a_{x_2} A_1 - a_{x_1} A_2}{bC_s \times (a_{y_1} a_{x_2} - a_{y_2} a_{x_1})} \times 100$$

Presentation of Data

9. If absorption curves are to be presented with an analytical method, it is recommended that one of the following systems be used, with the wavelength (in millimicrons) increasing linearly to the right:

$\log \epsilon$ or $\log a$ plotted against λ
A plotted against λ
$\epsilon \times 10^{-n}$ or a plotted against λ

where the symbols are as defined in Definitions E 131. Marking the analytical wavelengths and absorptivity values on the curve is suggested for clarity, or a separate table of analytical wavelengths and absorptivities may be used. (These data are helpful for others who may wish to use the method in a somewhat modified form.)

BIBLIOGRAPHY

Books

(1) M. G. Mellon, Editor, "Analytical Absorption Spectroscopy," John Wiley & Sons, N. Y.

(2) G. F. Lothian, "Absorption Spectrophotometry," Macmillan, N. Y., 2nd Ed. (1958).

(3) W. West, Editor, "Chemical Applications

of Spectroscopy," Vol. IX or Weissberger series, "Technique of Organic Chemistry," Interscience, N. Y. (1956).

(4) R. A. Friedel and M. Orchin, "Ultraviolet Spectra of Aromatic Compounds," John Wiley & Sons, N. Y. (1951).

(5) A. E. Gillam and E. S. Stern, "Electronic Absorption Spectroscopy," E. Arnold, London, 2nd Ed. (1957).

(6) H. M. Hershenson, "Ultraviolet and Visible Absorption Spectra: Index for 1930–1954," Academic Press, N. Y. (1956).

Reviews

(7) Annual or Biennial Reviews in Analytical Chemistry: E. J. Rosenbaum *21*, 16 (1949); *22*, 14 (1950); *23*, 12 (1951); *24*, 14 (1952); *26*, 20 (1954); R. C. Hirt, *28*, 549 (1956); *30*, 589 (1958) and *J. Chem. Ed. 29*, 301 (1952).

Articles

(8) R. Abbott, "Ultraviolet Spectrophotometric Techniques," *Applied Spectroscopy*, Vol. 4, No. 1, October, 1948.

(9) H. B. Klevens and J. R. Platt, "Ultraviolet Transmission Limits of Some Liquids and Solids," *Journal of the American Chemical Society*, Vol. 69, p. 3055 (1947).

(10) R. C. Hirt, F. T. King, and R. G. Schmitt, "Graphical Absorbance Ratio Method for Rapid Two-Component Spectrophotometric Analysis," *Analytical Chemistry*, Vol. 26, p. 1270 (1954).

(11) M. M. Graff, R. T. O'Connor, and E. L. Skau, "Purification of Solvents for Absorption Spectroscopy," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 16, p. 556 (1944).

(12) D. D. Tunnicliff, "Solvents for Ultraviolet Spectrophotometry," *Talanta*, Vol. 2, No. 4, p. 341 (1959).

REPORT OF COMMITTEE F-1
ON
MATERIALS FOR ELECTRON TUBES AND SEMICONDUCTOR DEVICES*

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices held three meetings during the year: at Skytop, Pa., on November 13 and 14, 1958; in Washington, D. C., on February 26 and 27, 1959; and in Boston, Mass., on June 18 and 19, 1959.

The committee sponsored a two-day symposium on the Cleaning of Electronic-Device Components and Materials, held in Philadelphia, October 13 and 14, 1958. The papers have been published by the Society as STP 246.

The membership of the committee totals 77 members, of whom 32 are classified as producers, 33 as consumers, and 12 as general interest members.

Committee Organization.—P. P. Prichett was appointed chairman of Subcommittee III on Strip, replacing C. W. Horsting. H. M. Pollack was appointed chairman of Subcommittee VI on Semiconductors, replacing H. C. Theurer.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee F-1 presented to the Society through the Administrative Committee on Standards the recommendations listed below. The recommendations were accepted by the Standards Committee on the dates indicated and the new and revised tentatives will appear in the 1959 Supplement to Book of ASTM Standards, Part 2.

Tentative Specification for:

Round Wire for Use as Grid Siderods in Electron Tubes (F 9 - 59 T) (Accepted April 13, 1959)

Tentative Recommended Practice for Testing:

Electron Tube Materials Using Reference Triodes (F 8 - 58 T) (Accepted November 13, 1958)

Revision of Tentative Method of Test for:

Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (F 205 - 56 T) (Accepted September 9, 1958)

Sag of Tungsten Wire (F 269 - 52 T) (Accepted September 9, 1958)

NEW TENTATIVE

The committee recommends for publication as tentative the Specification for Miniature Electron-Tube Leads as appended hereto.[1]

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

---

* Sixty-second Annual Meeting of the Society, June 21-26, 1959.

[1] The new tentative appears in the 1959 Supplement to Book of ASTM Standards, Part 2.

Standard Method for Bend Testing of Wire (Wire for Radio Tubes and Incandescent Lamps) (F 113 – 41):[2]

Revise as appended hereto.[3]

Standard Method of Testing Fine Round and Flat Wire for Electronic Devices (F 219 – 50):[2]

Title.—Change the title to read: "Standard Method of Testing Fine Round and Flat Wire for Electronic Devices and for Lamps."

Section 6(c).—Change to read: "(*c*) When using procedure A, the report shall include the maximum depth of cord reported to the nearest 0.5 mm."

Section 7(c).—Change to read as follows:

> (*c*) *Procedure B.*—This method is recommended for wires not readily measured in accordance with procedure A. A set of standard curves or standard wire specimens shall be established jointly by consumer and producer for visual comparison with samples representing maximum permissible curves and irregularities of various descriptions. These shall be mounted under glass in such a way that they cannot be disturbed or altered in straightness. Acceptance or rejection shall be based upon visual comparison of the unmounted specimens with the standards according to agreement between consumer and producer.

Section 8.—Change Paragraphs (*a*) and (*b*) to read as follows:

> (*a*) *Apparatus.*—Any standard testing machine that applies load at a constant rate of traverse is satisfactory, if agreed upon by manufacturer and purchaser. The capacity of the testing machine shall be such that all specimens fail at greater than 45 per cent of the capacity of the machine. The clamps used shall be such that there will be no slipping of or damage to the test specimen.
>
> (*b*) *Procedure.* — Tensile strength, yield strength, and elongation shall be determined at room temperature. The gage length of the specimen shall be 10 in. The initial load applied to the specimen before making the test shall be sufficient to keep the wire straight. The rate of traverse shall be 1 in. per min. The yield strength shall be defined as the stress at which the specimen exhibits an elongation of 1 per cent. If the specimen breaks within 0.5 in. of the clamps, the test shall be repeated.

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following standards, since the information contained in them has been incorporated in the new Tentative Specification for Round Wire for Use as Grid Siderods in Electron Tubes (F 9 – 59 T):

Standard Specifications for:

Round Chromium-Copper Wire for Electronic Devices (F 268 – 56).[2]

Standard Methods of Testing:

Wire for Supports Used in Electronic Devices and Lamps (F 157 – 50).[2]

EDITORIAL CHANGES

The committee recommends editorial changes as follows in the Tentative Specification for Aluminum Oxide Powder (F 7 – 58 T):[2]

Section 4(a).—Delete the second sentence, since ASTM Methods C 114 do not apply to this material nor are there spectrographic quantitative procedures.

Section 5.—Delete Paragraph (*a*), since ASTM Methods E 38 do not apply to nonmetallic materials, and reletter the subsequent paragraphs accordingly.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Cathode Materials (A. P. Haase, chairman):

The Data Section is working on the prove-in test for a tungsten-nickel cathode alloy. A major revision of the prove-in test method was accomplished during the year (ESI).

In the *Electrical Test Section*, the interface impedance report and history, written by William Buescher of Sylvania Electric Products Co., has been revised and approval is being asked for publication in a trade journal of broad coverage.

---

[2] 1958 Book of ASTM Standards, Part 2.

[3] The revised method appears in the 1959 Supplement to Book of ASTM Standards, Part 2.

The Reference Planar Diode Specification has been approved for letter ballot.

The *Task Group on Cathode Temperature Measurements* is investigating various methods such as optical pyrometry and thermocouples and infrared techniques.

The *Sublimation group* is devising an improvement of Methods F 278 covering Sublimation Characteristics of Metallic Materials by Electrical Resistance.

In the *Physical and Mechanical Test Section* a specification for the measurement of disk cathodes is being prepared. In addition, Methods of Testing Sleeves and Tubing for Radio Tube Cathodes (F 128) and Specifications for Nickel Alloy Cathode Sleeves for Electronic Devices (F 239) are being revised and will be reviewed at the next meeting.

The *Adherence Section* is working on cycling tests and numerous methods to evaluate adherence.

Subcommittee II on Insulators (C. M. Harman, chairman) expects to have a specification on mica within a year. Several methods of measuring the hole size in mica have been compared with the present hand-operated procedure. These are electrical and hydraulic means of introducing the pin gage.

Subcommittee III on Strip (C. W. Horsting, chairman) is completing work on the gas content of strip.

The major supplier of nickel has proposed a new chemical composition for nickel to be used for electronic cathodes. The specification for chemical composition of nickel to be used for electronic cathodes will be reviewed with other chairmen involved with this material.

Other active subjects are methods of measuring emissivity and consideration of specifications for the dimensions of cathode strip.

The nickel-clad steel specification is to be reviewed.

Subcommittee IV on Wire (D. R. Kerstetter, chairman).—Methods F 157 and Specification F 268 are being withdrawn as indicated earlier in this report. Specification F 175 is being kept in effect pending incorporation of the material in this specification within the Grid Siderod Specification (F 9).

Subjects which continue under investigation are the measurement of out-of-roundness, contamination of lead wire and siderod wire by the flame test, and the knot strength of miniature leads.

New matters to come under study are a proposed standard for heater wire sizes, and methods other than weighing for measurement of the diameter of wire.

Subcommittee V on Metallic and Non-Metallic Seals (V. J. DeSantis, chairman).—Specifications for iron-nickel alloys having compositions of 42, 46, and 52 per cent are being prepared. A series of tests is being run in five laboratories on the thermal expansion of these alloys.

Methods for measuring expansivity up to 1000 C are being evaluated, together with other tests for thermal conductivity, modulus of rupture at room temperature, 500 C, and 1000 C, and dielectric tests.

Specifications on ceramic materials are in process but will be preceded by methods of tests. A specification on tensile strength is still under consideration, and a specification on glass seals is ready for letter ballot.

Subcommittee VIII, Editorial (E. A. Thurber, chairman) reviewed the drafts of a specification for the disk cathode, and a recommended practice for growth of silicon single crystal test ingots. The reference planar diode specification is being re-edited.

Subcommittee IX on Materials Analyses (R. S. Kelly, chairman).—Wet analysis of the major elements in glass sealing alloys (nickel-cobalt-iron and nickel-iron) is under consideration. A better method for sulfur has been discussed.

Tests for the determination of tungsten in electronic nickel by the acid digestion cinchonine method, and the determination of magnesium in electronic nickel by the oxine (photometric) method, prepared by the subcommittee and approved by Committee F-1, have been referred to Committee E-3 on Chemical Analysis of Metals for promulgation.

Subcommittee X on Control of Contaminants (D. E. Koontz, chairman) was formed to develop test methods for processing materials such as air and water. They will also undertake tests as applied to parts.

This report has been submitted to letter ballot of the committee, which consists of 77 members; 51 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted in behalf of the committee,

S. A. STANDING,
Chairman.

STANTON UMBREIT,
Secretary.

TECHNICAL PAPERS

With Discussions

TENSION, COMPRESSION, AND FATIGUE PROPERTIES OF SEVERAL STEELS FOR AIRCRAFT BEARING APPLICATIONS*

By G. Sachs,[1] R. Sell,[1] and W. F. Brown, Jr.[2]

Synopsis

The static tensile and compressive properties, and the rotating-beam fatigue strength for several heats of SAE 52100 and three vacuum-melted tool steels were established at room and elevated temperatures and over a range of hardnesses. Static tensile properties reached a maximum at a certain hardness level depending on the alloy. The various heats of SAE 52100 had nearly equal elastic limits and yield strengths in both tension and compression. However, property differences between the three tool steels were observed in the static tests. The fatigue strength (10^8 cycles) of SAE 52100 remained essentially constant from about Rockwell hardness C 52 to 65, but was different for the various heats investigated, being highest for the vacuum-melted material. At a Rockwell hardness level of C 62 the 500 F static and fatigue strengths of the tool steels appeared superior to those of vacuum-melted SAE 52100 tested at 350 F.

On the basis of rather limited data, it appears that a correlation may exist between the rotating beam fatigue data for three heats of SAE 52100 steel and the median fatigue life of full scale bearings fabricated from the same heats.

Aircraft turbine bearings must operate under severe conditions of both load and temperature. An important problem is to increase both engine reliability and performance by increasing the bearing life of presently used alloys and by applying new alloys with greater strength at elevated temperature.

The common bearing steel is SAE 52100 heat treated in the range between Rockwell hardness C 58 and 62. A gradual improvement in SAE 52100 steel bearing life has been observed over the past 10 yr. As pointed out by Cobb (**1**),[3] this has probably resulted from improving the cleanliness of the steel. Thus, Barnes and Ryder (**2**) have shown that the minimum bearing life of SAE 52100 steel is greatly improved by vacuum melting. Similar beneficial effects of vacuum melting have been reported by Frith (**3**) and Styri (**4**) on the rotating-beam fatigue strength of this alloy, and by Ransom (**5**) on the fatigue strength of SAE 4340 steel.

The need for satisfactory bearing performance at temperatures exceeding the useful range of SAE 52100 steel has led to the consideration of vacuum melted tool steels (**1,3,6,7**). These steels exhibit strong secondary hardening characteristics, and Rockwell hardnesses of C 60 and above may be obtained by tempering at temperatures somewhat over 1000 F. Very little information exists regarding the static mechanical properties or the fatigue characteristics of such steels. However the properties in static bending have been reported by Grobe and Roberts (8) and by Hamaker, Strang, and Roberts (9). Values of bend strength increase with increasing hardness to over 600,000 psi at Rockwell hardnesses exceeding C 60.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Syracuse University Research Institute, Syracuse, N. Y.

[2] Lewis Research Center, Nat. Aeronautics and Space Administration (NASA), Cleveland, Ohio.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—CHEMICAL COMPOSITIONS OF MATERIALS TESTED, PER CENT.

Alloy	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Chromium	Vanadium	Tungsten	Molybdenum	Copper	Nickel
SAE 52100 steel[a] Electric furnace Heat No. 1	1.03	0.32	0.01	0.02	0.29	1.49	...	...	0.01	0.05	0.08
SAE 52100 steel[b] Electric furnace Heat No. 2	1.02	0.41	0.009	0.012	0.30	1.43	...	...	0.02	0.11	0.08
SAE 52100 steel[b] Electric furnace Heat No. 3	1.06	0.34	0.012	0.009	0.30	1.43	...	...	0.02	0.12	0.13
SAE 52100 steel[a] Induction vacuum	1.05	0.37	0.002	0.007	0.26	1.51	...	...	trace	0	0.02
Halmo alloy[a] Induction vacuum	0.59	0.31	0.005	0.007	1.10	4.79	0.51	...	5.22	...	...
M-1 alloy (AISI TMO)[a] Induction vacuum	0.80	0.25	0.004	0.007	0.32	3.76	1.15	1.53	8.54	0.01	0.07
MV-1 alloy (AISI M-50)[a] Induction vacuum	0.81	0.26	0.004	0.007	0.14	3.97	1.07	0.01	4.29	0.01	0.05

[a] Supplied by Crucible Steel Co., Syracuse N. Y.

[b] Supplied by Marlin Rockwell Corp., Jamestown, N. Y.

The direct evaluation of effects of melting practice, heat treatment, or alloy composition on bearing life are greatly complicated by the large number of tests necessary and the complex nature of the equipment. The beneficial effect of vacuum melting on both laboratory fatigue properties and bearing life suggest that there may be at least a qualitative relation between the results of these two types of tests. Furthermore, it has been claimed by various investigators that the bending fatigue endurance limit for heat-treated steels is directly proportional to the tensile strength. However, such a simple relation appears to be at best confined to low hardnesses and restricted to certain compositions (3,10,11). More recently, it has been suggested by Cohen and his co-workers (12,13) on the basis of rather meager evidence that both the tensile elastic limit and the endurance limit of low-alloy steels are similarly related to the hardness. It is suggested that the well-known decrease in elastic limit at high hardness levels is associated with a corresponding decrease in endurance limit. It might therefore be

TABLE II.—INCLUSION COUNT ACCORDING TO JK CHARTS (ASTM RECOMMENDED PRACTICE E 45 - 51).[a]

Alloy	Thin				Heavy			
	A	B	C	D	A	B	C	D
SAE 52100 steel Heat No. 1	2.0	1.5	3.0	2.0	1.0	1.0	1.0	1.5
SAE 52100 steel Heat No. 2	2.0	1.5	2.0	3.0	0.5	1.0	1.0	1.0
SAE 52100 steel Heat No. 3	1.5	3.0	2.0	2.0	0	1.0	1.0	1.5
SAE 52100 steel Induction vacuum	0.5	2.0[b]	0.5	1.0	0	0	0	0
Halmo alloy	0.5	2.0	0.5	2.0	0	0	0	0.5
M-1 alloy	0.5	1.5	0.5	2.0	0	0.5	0	0.5
MV-1 alloy	0	1.0	0	1.5	0	0	0	0

[a] Determined by Allegheny Ludlum Corp., Pittsburgh, Pa.; see footnote 4.

[b] One field, average rating = 1.0.

TABLE III.—HEAT TREATMENT SCHEDULES FOR SAE 52100 STEEL.[a]

SAE 52100 Steel	Rockwell Hardness, C Scale	Heat Treatment				
		Austenitizing Temperature, deg Fahr	Quench	First Temper		Second Hour Period
				Temperature, deg Fahr	Time, hr	Temper Temperature, deg Fahr
Heat No. 1	50	1535	Oil	320	0.5	775
	54			320	0.5	600
	58			320	0.5	525
	62			320	0.5	350
	65			300	0.33	...
Heat No. 2	50	1535	Oil	320	0.5	785
	54			320	0.5	700
	58			320	0.5	545
	62			320	0.5	365
	65			300	0.33	...
Heat No. 3	50	1535	Oil	320	0.5	785
	54			320	0.5	700
	58			320	0.5	545
	62			320	0.5	365
	65			300	0.33	...
Induction vacuum	50	1535	Oil	320	0.5	750
	54			320	0.5	625
	58			320	0.5	500
	62			320	0.5	340
	65			300	0.33	...

[a] Heat treated by Marlin Rockwell Corp., Jamestown, N. Y.

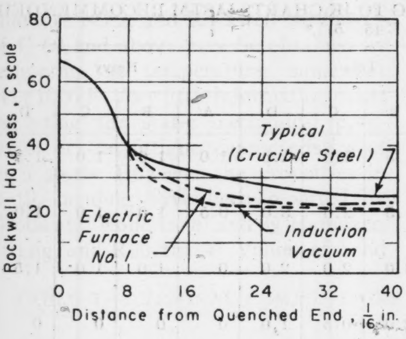


FIG. 1.—Hardenability of Two SAE 52100 Heats.

possible to estimate the influence of a given metallurgical variable on the bearing life by examining its effect on the tensile elastic limit, or perhaps on some other static mechanical property. To examine such a possibility, critically, data are needed for the static, compressive, and fatigue properties over a wide range of hardnesses.

The object of the present investigation was to establish the influence of heat-treated hardness level on the room temperature and 350 F static and fatigue properties of electric furnace melt and vacuum melt SAE 52100 steel and on the room temperature static properties of three tool steels. The static and fatigue properties of the tool steels were also investigated for one hardness level (C 62) at both room temperature and 500 F. In addition, a few tests were made to investigate the influence of minor variations in heat treatment on the 500 F fatigue strength of the tool steels. An attempt was made to determine the magnitude of first and second order residual stresses for all alloys as a function of hardness level. The results are analyzed to permit comparison of the mechanical properties of the tool steels with those of SAE 52100 steel and to explore the possibility of a relation between static and fatigue characteristics.

MATERIAL

Three electric furnace heats and one induction vacuum melted heat of SAE 52100 steel were investigated. Two of the electric furnace heats (Nos. 2 and 3) were available only in small quantities and represent material for which bearing life data was determined in another investigation (see Appendix II). Three induction vacuum melted tool steel compositions—Halmo alloy, M-1 alloy (AISI TMO), and MV-1 alloy (AISI M-50)—were also included. The composition and source of all these materials is given in Table I. With the exception of SAE 52100 steel, heats Nos. 2 and 3, all alloys were received as ½-in. diameter rod. These latter two heats were furnished in $\frac{9}{16}$-in. diameter.

The hardenability of the two principal heats of SAE 52100 steel is shown in Fig. 1 in comparison with typical data supplied by one steel company. The ASTM inclusion ratings[4] for the various alloys are given in Table II.

The various heat treating schedules employed are given in Tables III and IV. These include a series of experimental treatments designed to yield a range of Rockwell hardness from C 50 to 65. In addition, commercial heat treatments are included which yield a Rockwell hardness of about C 62 and are presumably representative of present-day bearing practice.

TEST PROCEDURE

The very high hardness level of the steels and the consequent limited plasticity required that special techniques be employed in the tension tests to minimize bending stresses caused by misalignment. Similar precautions were necessary in the compression test to obtain reliable values of the elastic properties and yield strengths. Specimens were rough machined from the bar stock to about 0.035 in. oversize, heat treated, and then finish machined by grinding and polishing.

---

[4] Recommended Practice for Determining the Inclusion Content of Steel (E 45 - 51), 1958 Book of ASTM Standards, Part 3, p. 529.

TABLE IV.—HEAT TREATMENT PROCEDURES FOR TOOL STEELS.[a, b]

Alloy	Type of Heat Treatment	Heat Treatment: Austenitizing Temperature, deg Fahr	Quench	First 2 Hours: Temper Temperature, deg Fahr	Second 2 Hours: Temper Temperature, deg Fahr	Rockwell Hardness C Scale Approximately
Halmo........	Experimental	2100	Air	1000	1145	50
					1115	54
					1090	58
					1060	62
					....	65
Halmo........	Commercial	2100	Oil	1000	1000	62
M-1...........	Experimental	2200	Oil	1000	1200	50
					1170	54
					1150	58
					1120	62
					....	65
M-1...........	Commercial	2200	Oil	1000	1000	63
MV-1.........	Experimental	2050	Oil	1000	1200	50
					1160	54
					1125	58
					1060	62
					....	65
MV-1.........	Commercial	2050	Oil	1000	1000	62

[a] Heat treated by Marlin Rockwell Corp., Jamestown, N. Y.
[b] All specimens preheated at 1500 F.

Tension and Compression Testing:

Tension specimens with highly concentric ground threads, Fig. 2, were employed in conjunction with an axial loading fixture (14). This technique permitted reliable tensile strengths to be determined at the highest hardnesses.

Compression tests were performed in a ball-bearing die set using heavy parallel ground compression anvils with a special centering device. As a further aid in reducing bending stresses, the edges of the accurately machined, square-ended compression specimens were slightly rounded. Room temperature checks of the alignment were made using three wire resistance gages spaced 120 deg apart on longitudinal elements of the specimens. Typical examples of elastic stress-strain curves from these three gages are shown in Fig. 3 for a tension and compression specimen. Perfect alignment would be indicated by identical curves from the three gages. As can be seen, this ideal condition is closely approached, the maximum bending stress

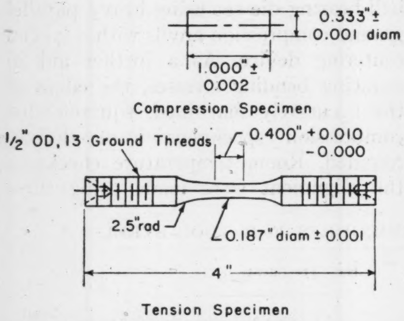

0.333" ± 0.001 diam
1.000" ± 0.002
Compression Specimen
1/2" OD, 13 Ground Threads
0.400" + 0.010 − 0.000
2.5" rad
0.187" diam ± 0.001
4"
Tension Specimen

FIG. 2.—Tension and Compression Specimens.

Note.—Threads to be concentric with centerline of test section within 0.0005 in.

being approximately 1 to 2 per cent of the average tensile stress and 2 to 4 per cent of the average compressive stress.

For elevated temperature testing, fundamentally the same techniques were employed. In the case of compression tests, the anvils were extended inside of a split furnace. For both tension and compression tests, the temperature variation along the gagelength was within ±2 F.

Fatigue Tests:

The specimens employed at room temperature were of standard design,

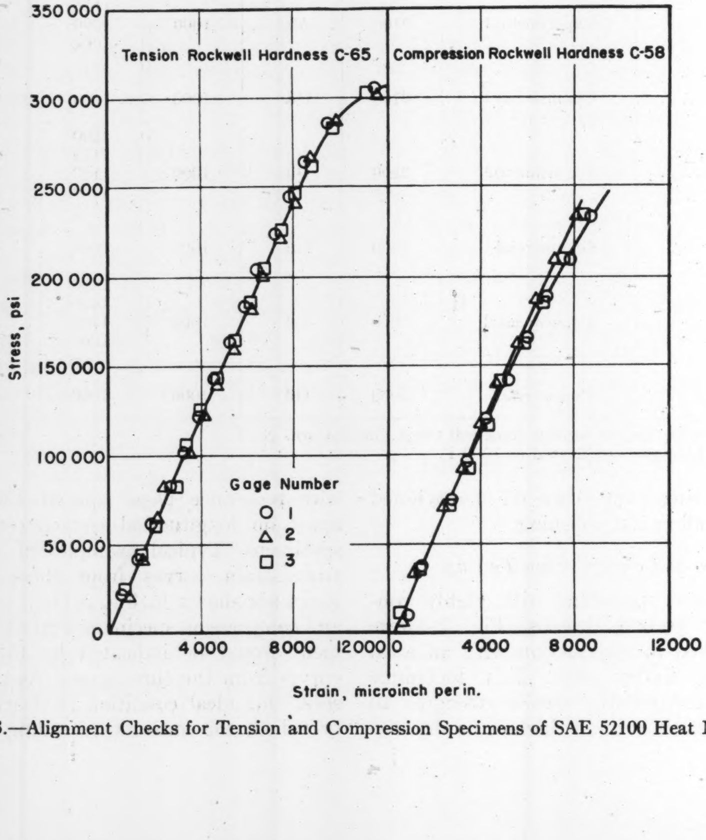
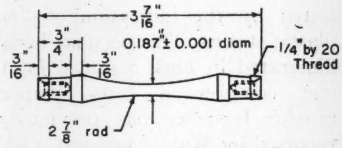
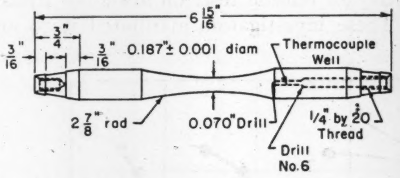


FIG. 3.—Alignment Checks for Tension and Compression Specimens of SAE 52100 Heat No. 1.



Room Temperature Fatigue Specimen



Elevated Temperature Fatigue Specimen

FIG. 4.—Room and Elevated Temperature Rotating Bending Fatigue Specimens.

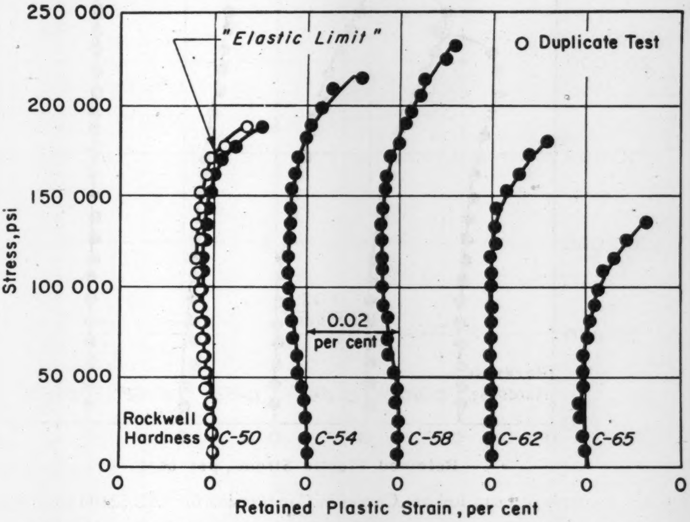


FIG. 5.—Example of Load Release Curves in Tension for SAE 52100 Heat No. 3.

Fig. 4, and finish polished in the longitudinal direction.[5] Standard R. R. Moore rotating-bending fatigue machines were employed for testing these specimens.

For tests at elevated temperature, the specimens, Fig. 4, were provided with a 1¾-in. cylindrical length at each end. This design permitted the installation of a tubular electric-resistance furnace over the test section. In order to accommodate the furnace, the fatigue machines were modified to increase the distance between the spindles by 3½-in. A water-cooled jacket was fixed between the face of each spindle housing and the furnace end. Temperature control was effected by means of a thermocouple inserted into a bore through one end of the specimen which terminated ⅞-in. from the minimum diameter. The temperature variation at the test section did not exceed ±10 F.

Strain Measurements:

The determination of elastic limit in tension and compression at room temperature was by a "load release" method which has been employed previously by other investigators. For this method an SR-4, Advance wire strain gage was Duco-cemented to the specimen and the load was increased in increments, unloading after each increment. The permanent set (plastic strain) after each unloading was recorded. Theoretically, the elastic limit would be represented by the highest stress value not producing plastic strain. The curve resulting from a plot of the stress against permanent set should be vertical (zero plastic strain) up to the elastic limit and then exhibit a continuous deviation in the direction of increasing plastic strain. Curves of this nature have been reported by McAdam and Mebs (15) using highly sensitive mechanical strain gages. Unfortunately, the use of SR-4 gages introduces difficulties. These are manifested by the indication of "reverse" plastic strains. This peculiar behavior is illustrated in Figs. 5 and 6 for tension and compression tests respectively. Similar behavior has previously been reported by Muir, Averbach, and Cohen (12) for tension tests on low-alloy steels. These investigators attributed it to an effect characteristic of the metal itself. However, it may be explained by certain effects characteristic of the wire strain gage. Thus, it has been shown by Campbell (17,18) that Duco bonded Advance wire gages show deviations from strains measured on the same specimen with a Tuckerman extensometer. These deviations are such that the actual strain

---

[5] Finish polishing was done in three steps using wet 80, 280, and 500 grit paper in this order.

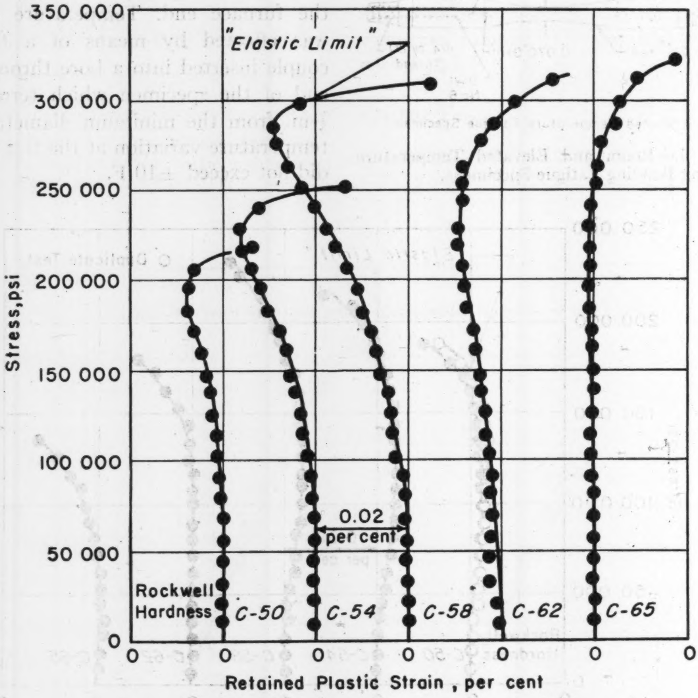


FIG. 6.—Example of Load Release Curves in Compression for SAE 52100 Heat No. 3.

exceeds the indicated strain. This effect increases with the applied strain and at strains in the plastic range (above about 2.0 per cent), the errors are in excess of 0.10 per cent strain. When gages showing such deviations are unloaded a zero shift is observed which is in the opposite direction to the applied strain and increases with the applied strain. Thus, on unloading the wire gage will indicate a permanent set in a direction opposite to the preceeding strain.[6]

These zero shifts in combination with residual stresses could produce the peculiar variation in the amount of reverse strain with hardness and its different magnitude in tension and compression. Therefore, no real quantitative value of the elastic limit can be derived from the reported load release data. However, for purposes of comparison, an "elastic limit" is defined as the highest stress resulting in an indicated zero plastic strain.

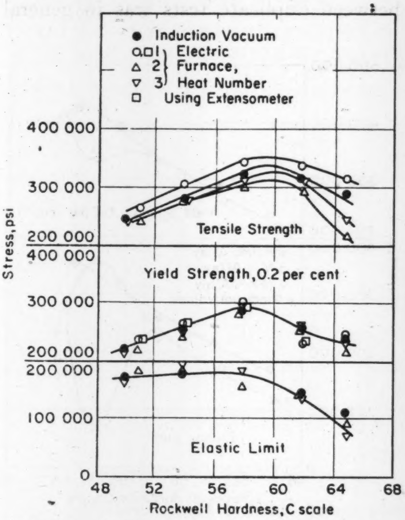


FIG. 7.—Room Temperature Tension Characteristics for Various Heats of SAE 52100 Steel.

Values shown are average of two or more tests unless indicated.

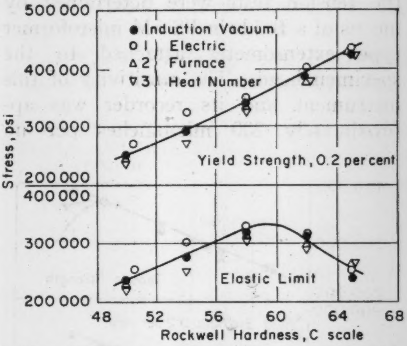


FIG. 8.—Room Temperature Compression Characteristics for Various Heats of SAE 52100 Steel.

Values shown are average of two or more tests unless indicated.

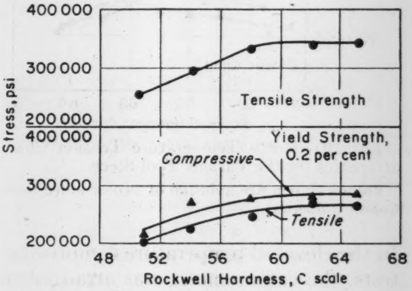


FIG. 9.—350 F Tension and Compression Characteristics of SAE 52100 Heat No. 1.

Values shown are average of two or more tests unless indicated.

The 0.2 per cent yield strength was determined on the same specimens used for the load release curves by extending the SR-4 readings slightly beyond 0.2 per cent strain. This rather unconventional manner of determining yield strength was checked by use of an extensometer. Yield strengths determined by these two methods were essentially identical, as might be expected, considering the flatness of the flow curve in the yield region.

---

[6] Direct experimental evidence is presented by Newton (ASTM BULLETIN, Jan., 1959, p. 423) in a paper published after this manuscript was written. It is shown that the reverse strains are not a real property of the specimen but are associated with zero shift in the Duco cemented wire gage.

At elevated temperature, strains in the tension tests were determined by means of a Baldwin PS 5M microformer type extensometer attached to the specimen heads. The sensitivity of this instrument and its recorder was approximately 200 microinches per in. In the elevated temperature compression tests, the extensometer was arranged to sense the movement of the die set plattens. No attempts were made to determine elastic properties at elevated temperatures.

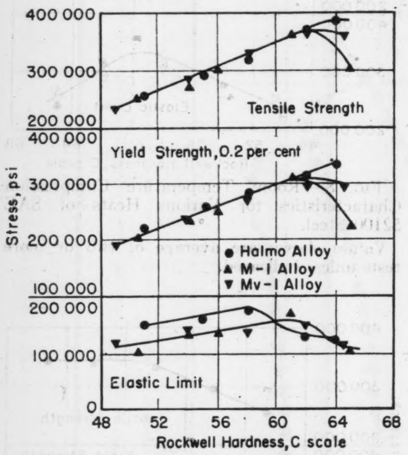


Fig. 10.—Room Temperature Tension Characteristics for the Various Tool Steels.

Values shown are average of two or more tests unless indicated.

TEST RESULTS

The results obtained establish the influence of Rockwell hardness C 50 to 65 on the room temperature static tensile and compressive properties of several heats of SAE 52100 steel and three tool steels. In addition, the 350 F static properties of an electric furnace melt of SAE 52100 are presented as a function of hardness. Static properties at 500 F are shown for the three tool steels heat treated to C 62 by a typical commerical heat treatment. In plots of the static properties, the points shown are the average of at least two tests unless otherwise indicated. The spread between duplicate tests was in general less than ±5 per cent with a few elastic limit values differing by ±15 per cent.

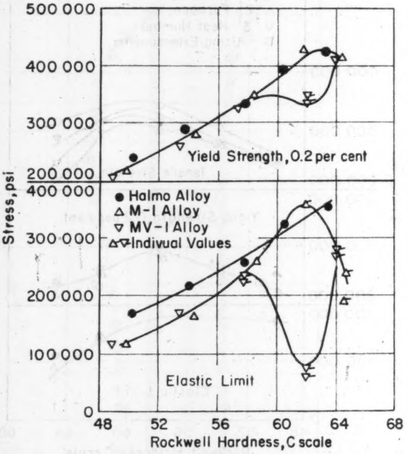


Fig. 11.—Room Temperature Compression Characteristics for the Various Tool Steels.

Values shown are average of two or more tests unless indicated.

Fatigue properties at room temperature are established as a function of hardness level (C 50 to 65) for several heats of SAE 52100 steel and for the three tool steels commercially heat treated to C 62. Fatigue data at 350 F are also presented for an electric furnace melt and an induction vacuum melt of SAE 52100 at several hardness levels. Fatigue properties at 500 F for the three tool steels are presented for one hardness level (C 62) obtained by a commercial and experimental heat treatment. Since the number of fatigue specimens available were quite limited, the majority of tests were confined to the lower stress levels in order to best estimate the fatigue strength between 5 × 10^7 and 10^8 cycles. The accuracy of the fatigue strength at 10^8 cycles is in no case better than ±10,000 psi.

TABLE V.—EFFECTS OF ELEVATED TEMPERATURE ON MECHANICAL PROPERTIES OF VACUUM-MELTED BEARING STEELS AND SAE 52100 VACUUM MELT AT ROCKWELL HARDNESS C 62.

Alloy	Temperature, deg Fahr	Tensile		Compressive Yield, psi	Fatigue Strength, 10^7 cycles, psi
		Strength, psi	Yield, psi		
52100 steel	Room	340 000	240 000	400 000	130 000
	350	340 000	260 000	290 000	95 000
Halmo[a]	Room	370 000	310 000	410 000	140 000
	500	335 000	310 000	340 000	110 000
M-1[a]	Room	370 000	310 000	420 000	130 000
	500	350 000	300 000	355 000	110 000
MB-1[a]	Room	370 000	295 000	340 000	125 000
	500	335 000	300 000	340 000	100 000

[a] Commercial heat treatment, see Table IV.

Static Properties of SAE 52100 Steel:

The various heats of SAE 52100 steel possess only slight differences in their room temperature elastic limits and yield strengths (Figs. 7 and 8) in both tension and compression over the entire hardness range. Tensile strengths exhibited somewhat greater differences, particularly at Rockwell hardnesses above C 58. Surprisingly the highest tensile strengths are observed for an electric furnace melt and not as might be expected for the vacuum melt.

The influence of hardness on all room temperature static strength properties except the compressive yield strength is similar in that a nearly linear increase in strength is observed up to C 58, the strength values then continuously decrease with increasing hardness. In contrast, the compressive yield strength increases linearly to the highest hardness.

According to Fig. 9, the 350 F static properties of electric furnace melt SAE 52100 increase with hardness up to about C 58 and then remain essentially constant.

It is noted at both room temperature and 350 F that the static properties in compression are distinctly higher than those in tension.

Static Properties of the Tool Steels:

The room temperature static properties of the tool steels are shown in Figs. 10 and 11 as a function of the hardness. Some rather complex and unexpected trends are observed. While these are clearly indicated by the available data, additional tests would be necessary to establish them as general behavior for the alloys in question.

With the exception of the elastic limits, the three tool steels differ little in their static properties except at the highest hardnesses. At hardnesses above about Rockwell hardness C 62 the Halmo alloy appears to possess the highest and M-1 alloy the lowest tensile ultimate and yield strength. The compressive yield strengths at high hardnesses are essentially identical with the exception of the anomalous behavior of MV-1 alloy at C 62 hardness. The elastic limits in both tension and compression exhibit unexpected behaviors which render a comparison of the steels regarding this property difficult. Generally, it would appear that Halmo alloy exhibits elastic limits higher than either M-1 or MV-1 alloys at hardnesses less than about C 60. At higher hardnesses, irregularities in the elastic limits are observed. However, Halmo alloy appears to yield consistently high values.

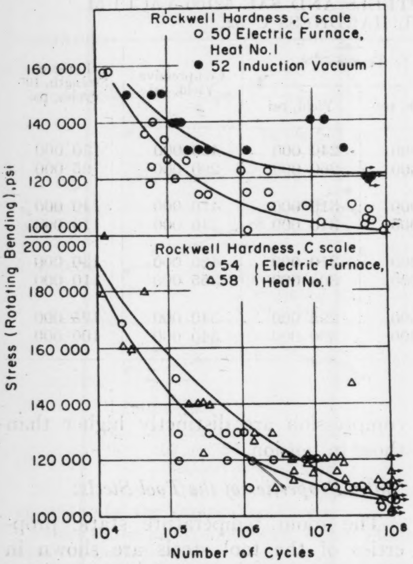


FIG. 12.—Room Temperature *S-N* Curves for Two Heats of SAE 52100 Steel at Several Hardness Levels.

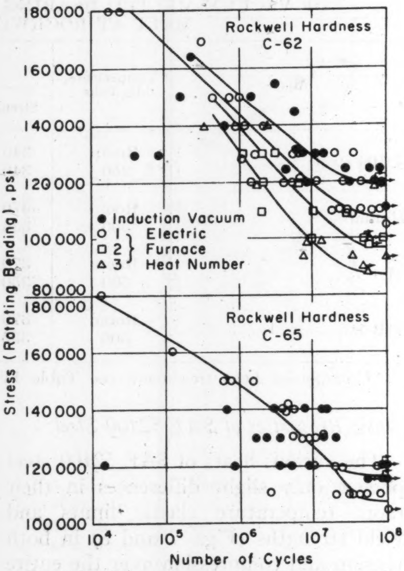


FIG. 13.—Room Temperature *S-N* Curves for Several Heats of SAE 52100 Steel Tested at Two Hardness Levels.

The influence of hardness on the room temperature tensile yield and ultimate strengths appears reasonably well established. These strength values increase nearly linearly with hardness up to a maximum at about C 62 with the exception of Halmo alloy which exhibits a continuous linear increase to the highest hardness. The compressive yield strength shows a similar linear increase up to a hardness of about C 62 for Halmo alloy and M-1 alloy. For MV-1 alloy an unexpected minimum was observed at Rockwell hardness C 62. Generally, the elastic limits in both tension and compression increased with hardness up to a hardness between C 58 and 62, depending on the alloy. At higher hardnesses, these values exhibit complex trends (particularly for MV-1 alloy) which may or may not be real. As might be expected, a decrease in elastic limit is observed with further increasing hardness for tension tests. For compression tests, a similar indication of a maximum is observed for Halmo and MV-1 alloys while the elastic limit for MV-1 alloy passes through a minimum.

The 500 F tension yield and ultimate strength and the compressive yield strength are given in Table V. It will be noted that there is little difference between the three tool steels at a Rockwell hardness of C 62 regarding these static properties.

As was observed for SAE 52100 steel, the room temperature and elevated temperature static properties of the tool steels in compression exceed those in tension.

Fatigue Properties of SAE 52100 Steel:

Room temperature *S-N* curves at various hardnesses are given in Figs. 12 and 13. It will be noted that distinct endurance limits are not established. The results for the several heats are compared in Fig. 14 which shows the fatigue strength at 10^8 cycles plotted against the hardness. The induction vacuum melt appears to be superior to the electric furnace melts. The influence of hardness on either the electric furnace or vacuum melted alloy is rather small, the fatigue strength being essentially constant from Rockwell hardness C 54 to C 65.

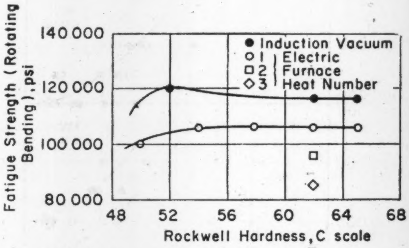


FIG. 14.—Comparison of Room Temperature Fatigue Strengths at 10^8 Cycles for the Various Heats of SAE 52100 Steel as a Function of Hardness Level.

At 350 F only a limited number of tests were made (Fig. 15), and the induction vacuum melt was evaluated only at Rockwell hardness C 62. The influence of hardness at 350 F can be estimated by comparing the 350 F fatigue strength at 10^8 cycles with that obtained at room temperature. Thus according to Fig. 16 the 350 F fatigue strength remains essentially constant up to about Rockwell hardness C 56 and then decreases slightly to a lower constant value at hardnesses above about C 60.

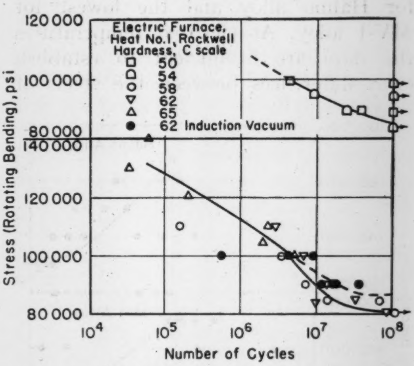


FIG. 15.—350 F *S-N* Curves for Two Heats of SAE 52100 Steel Tested at Several Hardness Levels.

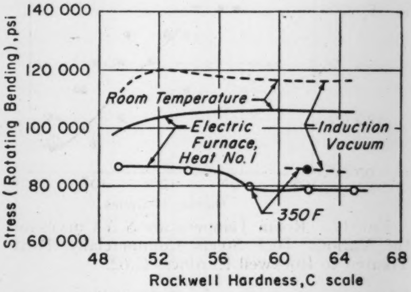


FIG. 16.—Comparison of Room Temperature and 350 F Fatigue Strengths at 10^8 Cycles for an Electric Furnace and Induction Vacuum Heat of SAE 52100 Steel.

Fatigue Properties of Tool Steels:

The room temperature and 500 F *S-N* curves are shown in Figs. 17 and 18 for the three tool steels heat treated to Rockwell hardness C 62. Because of the limited number of tests and excessive scatter fatigue strengths at 10^8 cycles are not well established. At room temperature, the highest fatigue strengths (10^7 to 10^8 cycles) are obtained for Halmo alloy and the lowest for MV-1 alloy. At elevated temperatures the data are insufficient to establish clear differences between the steels or between the commercial and experimental heat treatment. The most consistent results both in regard to scatter and agreement between the two heat treatments was obtained with Halmo and M-1 alloys which both appear to possess nearly equal fatigue strengths. For MV-1 alloy, the 500 F fatigue strength appears to be highest for the commercial heat treatment which yields strength values about equal to the other two steels.

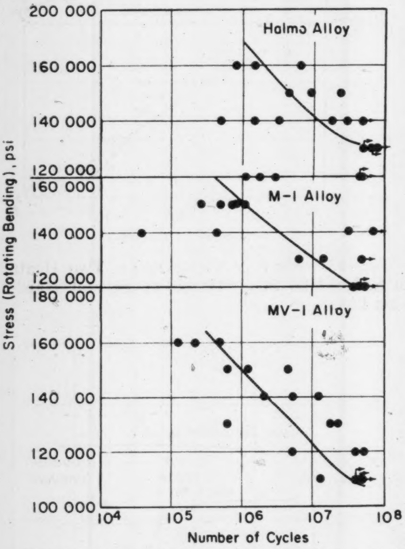


FIG. 17.—Room Temperature *S-N* Curves for the Various Tool Steels Commercially Heat Treated to Rockwell Hardness C 62.

DISCUSSION OF RESULTS

In the following section the various observed mechanical properties will be further discussed in light of the residual stress measurements given in Appendix I and compared with previously published information. Further, the possibility of a relation between static and fatigue properties will be explored.

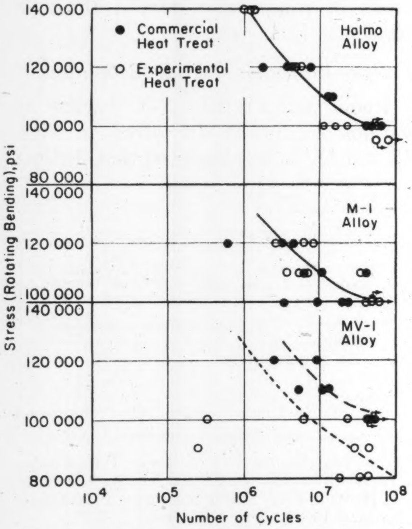


FIG. 18.—500 F *S-N* Curves for the Various Tool Steels Heat Treated to Rockwell Hardness C 62 by Two Procedures.

Influence of Residual Stresses:

Maxima in the tensile and compressive elastic limits and in the tensile yield strength when plotted against the hardness have been observed in many investigations (11) of low-alloy heat-treated steels. Similar behavior has also been reported for the bend yield strength of tool steels (8,9) tempered to very high hardness. In addition it has been previously reported (11,19) that the compressive yield strength is higher than the tensile yield and that this difference increases with the hardness. The magnitude of these effects appears to depend on the heat treating practice which sets the residual stress pattern.

Residual stresses in the investigated steels are reported in Appendix I (see Figs. 20 and 21). These data indicate tensile strains in the (211) planes parallel to the surface as well as high microstrains, both of which increase with hardness. Such results are not unexpected and may explain in part the shape of the trend curves when the static properties are plotted against the hardness, as well as the difference in strengths observed between tension and compression. The microstrains cause early deviation of the stress strain curve from linearity and tend to depress the elastic limit and yield strength. The first order residual stresses arise primarily from compressive plastic surface deformations occurring during the quenching cycle. It may be assumed that on subsequent testing these surface fibers are subject to a type of "Bauschinger effect." In tension tests both the microstrains and this Bauschinger effect combine to depress the flow curve at small plastic strains. These effects are reduced by tempering and consequently are most pronounced at the highest hardnesses (lowest tempering temperatures).

Thus, the static tensile yield and elastic limits pass through a maximum at some high hardness level. A maximum in the tension ultimate is observed at approximately the same hardness since the fracture ductilities are so low (less than 2 per cent) that neither the Bauschinger effect nor the microstrains are completely eliminated by plastic flow. In compression, the Bauschinger effect is absent and consequently the elastic limits and yield strengths are higher than in tension and the maxima are absent.

Regarding the fatigue strength, it might be expected to increase continuously with hardness in the absence of other effects. However, as seen from Fig. 14, it remains essentially constant above Rockwell hardness C 52. The same phenomena discussed above may be associated with this behavior. The fatigue test is, however, much more complex than the static tests, and it is not possible to make meaningful conclusions from the limited data available. The literature (**20,21,22**) only serves to emphasize the complexity of the problem when residual stresses are introduced primarily by heat treatment or machining.

Comparison of Fatigue Strengths for SAE 52100 Steel:

The present result that the fatigue strength of SAE 52100 steel does not decrease at high hardnesses is supported by less extensive data from several previous investigations (**3,4,23**). This behavior is in contrast to the results of Garwood, Zurburg, and Erickson (**24**) who report the endurance limit for a number of low-alloy steels passes through a maximum at high hardness levels. Apparently no generalizations can be made regarding the dependence of fatigue strength on the hardness at high hardness levels.

Values of rotating-beam room temperature fatigue strength at 10^8 cycles for air-melted SAE 52100 steel have been reported previously (**3,4,23**). At bearing hardness levels, these vary considerably. For example, Styri (**4**) reports about 120,000 psi for this alloy quenched and tempered to Rockwell hardness C 63.5, while Frith (**3**) gives a value as low as 80,000 psi. Generally, the results from those investigations (**3,4**) where a considerable amount of data is available at 10^8 cycles appear to indicate that no definite endurance limit exists for SAE

52100 steel heat treated to high hardness levels.

These large variations in fatigue strength cannot be simply explained. Fatigue properties depend in a complex manner on a number of factors including the residual stresses resulting from quenching or machining, the steelmaking practice, and minor variations in the chemistry. As previously discussed, the effects of residual stresses are as yet obscure. Regarding the steelmaking practice, it has been reasonably well established in this and in other investigations (3,4) that vacuum melting increases the fatigue strength. According to Frith (3) the fatigue strength of open-hearth heats is higher than electric furnace melts. The explanation for these differences produced by variations in the steel-making practice may lie in the resulting inclusion and residual element content. Vacuum melting is known to reduce both residuals and inclusions. Thus, the vacuum melted SAE 52100 steel has a lower residual element content (Table VI) and a lower inclusion count (Table II) than the air melted heats. Furthermore, it is noted that there are only small differences in the inclusion counts of the three air-melted heats but that the room temperature fatigue strength of these three heats increases with a decrease of residual elements. These observations regarding the inclusion content are

TABLE VI.—INDICATIONS FOR RETAINED AUSTENITE AND CARBIDES.

Alloy	[a]	Rockwell Hardness				
		C 50	C 54	C 58	C 62	C 65
52100 steel Heat No. 1	*c*	. . .	. . .	. . .	. . .	trace
	A	. . .	. . .	. . .	trace	trace
52100 steel Induction vacuum	*c*	. . .	. . .	. . .	. . .	. . .
	A	. . .	. . .	. . .	trace	trace
Halmo	*c*	. . .	. . .	trace	weak	weak
	A	. . .	. . .	trace	weak	weak
M-1	*c*	strong	strong	strong	strong	strong
	A	. . .	trace	trace	trace	strong
MV-1	*c*	. . .	. . .	. . .	. . .	. . .
	A	. . .	weak	weak	weak	weak

[a] *c* = Carbide indication.
A = Austenite indication.

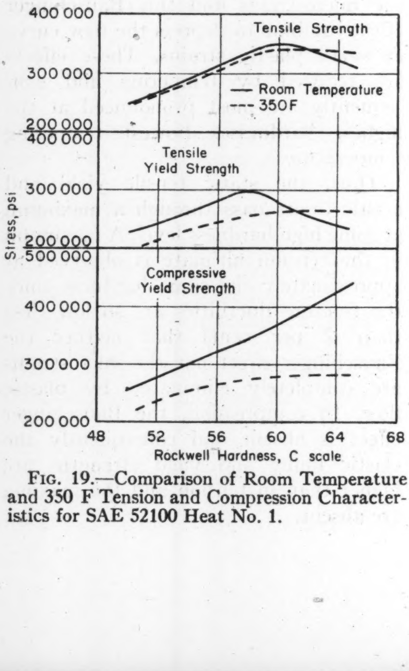


FIG. 19.—Comparison of Room Temperature and 350 F Tension and Compression Characteristics for SAE 52100 Heat No. 1.

difficult to account for on a quantitative basis. Thus, microexamination of fractures in this investigation failed to reveal any significant difference in their inclusion patterns. This observation is in accord with the results of Hyler, Tarasov, and Favor (25)[7] who report no positive correlation between fatigue strength and the size or location of inclusions in an SAE 52100 type steel. In contrast, Stulen, Cummings, and Schulte (26)[6] report the fatigue strength is strongly dependent on the size and location of inclusions.

Of interest in this connection is the work of Butler, Bear, and Carter (7) and Bear and Butler (28). It is shown that the failure of balls in simulated service tests occurs preferentially at the location of inclusions. Furthermore, when the rolling direction was so controlled that the maximum stress was perpendicular to the fiber, the ball fatigue life was considerably reduced. These observations point to the importance of inclusions in ball-bearing applications.

Effects of Elevated Temperatures:

A comparison of the static properties of electric furnace SAE 52100 steel as a function of the hardness is shown in Fig. 19 for room temperature and 350 F tests. The effect of increasing the test temperature conforms to the previously established behavior of low-alloy steels (11). Thus, up to a hardness of about C 58 the tensile strength is little reduced by the increase in test temperature, while the yield strength in tension or compression at 350 F is 10 to 15 per cent lower than at room temperature. At hardnesses up to about C 58 the steel is quite stable. However, at a hardness of C 62 and above, the 350 F test temperature is close to or exceeds the tempering temperature and the structure is unstable. This instability permits a reduction in both microstrains and the previously suggested Bauschinger effect. The result is a slightly higher tensile yield and ultimate strength at 350 F compared with room temperature and an increase in the spread between the compressive yield strengths at these two temperatures.

Raising the test temperature from room to 350 F results in approximately 15 per cent loss in SAE 52100 steel fatigue strength up to Rockwell hardness C 54 (see Fig. 16). At higher hardnesses, the test temperature approaches or exceeds the tempering temperature and specimens soften during the test. As might be expected, the 350 F fatigue strength then decreases to a nearly constant value at hardnesses above C 58. In this range it is about 25 per cent lower than the room temperature fatigue strength.

The 500 F properties of the tool steels in comparison with those at room temperature are given in Table V for a Rockwell hardness of C 62. At 500 F the tool steels should be quite stable and the effect of test temperature would be expected to parallel that described above for SAE 52100 steel tempered to hardnesses less than C 58. However, somewhat different behaviors are observed. Raising the test temperature to 500 F reduces the tensile strength of the three tool steels by about 10 per cent, the compressive yield strength by about 15 per cent, the fatigue strength by as much as 20 per cent (Halmo alloy) and has essentially no effect on the tensile yield strength.

It is interesting to compare the 350 F mechanical properties of vacuum melt

---

[7] These investigators examined fractured specimens. Negative conclusions were reached by Starkey, Marco, and Gatts (27) for SAE 4330, 4340, and 4350 steels on the basis of a statistical analysis of fatigue data and the SAE inclusions count on unfractured specimens.

SAE 52100 with those of the tool steels at 500 F. According to Table V the static tensile strength of the tool steels at 500 F is essentially equal to that of vacuum melt SAE 52100 at 350 F. However, the tool steels are definitely superior in regard to their tensile yield, compressive yield and the fatigue strength.

Comparison of Static and Fatigue Properties:

Recently it has been proposed (12,13) on the basis of limited data that the endurance limit and the elastic limit of hardened alloy steels may be similarly related to the hardness. Examination of these data reveal that there is very meager evidence to support the hypothesis. The results of the present investigation do not confirm the proposed relationship. Thus, referring to Figs. 7 and 8, the room temperature tensile and compressive elastic limits of several SAE 52100 steel heats decrease at hardnesses above about C 58. However, the room temperature fatigue strength for the same heats, Fig. 14, remains essentially constant between Rockwell hardness C 52 and 65. It may be argued that true elastic limits were not established. However, the elastic limits previously reported (12,13) were obtained by essentially the same method used in this investigation and therefore may be subject to the same deficiencies.

A related observation is that vacuum melting definitely improves the room temperature and 350 F fatigue strength of SAE 52100 steel (see Figs. 14 and 15) but has little influence on the static properties (see Figs. 7 and 8).

Apparently for SAE 52100 steel the factors which control the static and fatigue properties are fundamentally different and neither are as yet well understood.

CONCLUSIONS

The complex nature of the phenomena observed and the limited number of tests performed render difficult the formulation of quantitative conclusions regarding the influence of alloy composition on the strength values. Furthermore, it is not considered that true elastic limits were determined nor were the 500 F fatigue strengths of the tool steels well established. However, for practical purposes the following conclusions appear substantiated:

1. The room temperature tensile ultimate, tensile yield and compressive yield strengths of SAE 52100 steel and the three tool steels increase nearly linearly with hardness when heat treated to yield hardnesses between about Rockwell hardness C 50 and 58. At a given hardness in this range only small strength differences are noted among the various steels.

2. At higher hardnesses the above relations at room temperature are generally maintained only for the compressive yield strength. The tensile static properties with the exception of Halmo alloy exhibit a maximum at a certain hardness and then decrease with further increases in hardness. Thus, at hardnesses above about Rockwell C 60 Halmo alloy appears to exhibit the highest tensile strength characteristics.

3. The room temperature fatigue strength (10^7 to 10^8 cycles) at a Rockwell hardness of C 62 is nearly identical within the limits of scatter for vacuum melt SAE 52100 and the three tool steels.

4. At a Rockwell hardness level of C 62 the tool steels tested at 500 F possess static and fatigue properties superior to those of SAE 52100 steel tested at 350 F.

5. The room temperature fatigue strengths of SAE 52100 steel at Rockwell

hardnesses between C 50 and C 65 is improved by induction vacuum melting.

6. The static properties of SAE 52100 steel are not improved by induction vacuum melting.

7. No relation was found between any of the measured static properties and the fatigue characteristics.

8. On the basis of rather limited data, it appears that a correlation may exist between the rotating beam fatigue data for three heats of SAE 52100 steel and the median fatigue life of full scale bearings fabricated from the same heats. (See Appendix II.)

Acknowledgments:

The authors are indebted to the National Advisory Committee for Aeronautics (NACA) for support of this project (Contract No. NAw 6521) and to various members of the NACA Subcommittees on Powerplant Materials and on Lubrication and Wear for their comments during the course of the investigation. Thanks are also due to M. H. Jones of the National Aeronautics and Space Administration (NASA), Lewis Research Center for his analysis of the SR-4 strain gage data and to V. Weiss of Syracuse University for the X-ray determinations reported in Appendix I.

REFERENCES

(1) L. D. Cobb, "Effect of Vacuum Melting on Bearing Steels," Soc. Automotive Engrs. preprint, March, 1957.

(2) G. C. Barnes and E. A. Ryder, "A Look at Some Turbine Bearing Problems," *Preprint No. 693*, Soc. Automotive Engrs. (1956).

(3) P. H. Frith, "Fatigue Tests on Rolled Alloy Steels Made in Electric and Open Hearth Furnaces," *Special Report No. 50*, Iron and Steel Inst., London (1954).

(4) H. Styri, "Fatigue Strength of Ball Bearing Races and Heat Treated 52100 Steel Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 682 (1951).

(5) J. T. Ransom, "The Effect of Inclusions on the Fatigue Strength of SAE 4340 Steels," *Transactions*, Am. Soc. Metals, Vol. 46, p. 1254 (1954).

(6) B. B. Muvdi, G. Sachs, and E. P. Klier, "Axial Load Fatigue Properties of High Strength Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 655 (1957).

(7) R. H. Butler, H. R. Bear, and T. L. Carter, "The Effect of Fiber Orientations on Ball Failures Under Rolling Contact Conditions," *Technical Note No. 3933*, Nat. Advisory Committee for Aeronautics, Feb. 1957.

(8) A. H. Grobe and G. A. Roberts, "The Bend Test for High Speed Steel," *Transactions*, Am. Soc. Metals, Vol. 40, p. 435 (1948).

(9) J. C. Hamaker, V. C. Strang, and G. A. Roberts, "Bend-Tensile Relationships for Tool Steels at High Strength Levels," *Transactions*, Am. Soc. Metals, Vol. 49, p. 550 (1957).

(10) P. H. Frith, "Fatigue of Wrought High-Tensile Alloy Steels," International Conference on Fatigue of Metals, Institution of Mechanical Engrs., London (1956).

(11) G. Sachs, "Survey of Low Alloy High Strength Steels Heat Treated to High Strength Levels," Part 2: Fatigue, *Technical Report 53-254*, Wright Air Development Center (1954).

(12) H. Muir, B. L. Averbach, and M. Cohen, "The Elastic Limit and Yield Behavior of Hardened Steels," *Transactions*, Am. Soc. Metals, Vol. 47, p. 380 (1955).

(13) C. H. Shih, B. L. Averbach, and M. Cohen, "Some Effects of Silicon on the Mechanical Properties of High Strength Steels," *Transactions*, Am. Soc. Metals, Vol. 48, p. 86 (1956).

(14) M. H. Jones and W. F. Brown, Jr., "An Axial Loading Creep Machine," ASTM BULLETIN, No. 211, Jan. 1956, p. 53 (TP25).

(15) D. J. McAdam, Jr. and R. W. Mebs, "Tensile Elastic Properties of Typical Stainless Steels and Nonferrous Metals as Affected by Plastic Deformation and by Heat Treatment," *Report No. 696*, Nat. Advisory Committee for Aeronautics (1940).

(16) "Physical and Electrical Properties of

Fine Resistance Wire," *Technical Bulletin No. 157*, Driver-Harris Co., N. J.

(17) W. R. Campbell, "Performance Tests of Wire Strain Gages, Part I Calibration Factors in Tension," *TN 954* (1944); "Part II Calibration Factors in Compression," *TN 978* (1945); "Part III Calibrations at High Tensile Strains," *TN 997* (1945); National Advisory Committee for Aeronautics Technical Notes.

(18) W. R. Campbell, "Errors in Indicated Strain for a Typical Wire Strain Gage caused by Prestraining, Temperature Changes and Weathering," *Technical Note TN 1011*, National Advisory Committee for Aeronautics (1946).

(19) L. J. Klingler, C. C. Chow, and G. Sachs, "Flow and Fracture Characteristics of a Die Steel at High Hardness Levels," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 185, p. 927 (1949).

(20) H. Sigwart, "Influence of Residual Stresses on the Fatigue Limit," International Conference on the Fatigue of Metals, Institution of Mechanical Engrs., London (1956).

(21) R. L. Matison: "Fatigue Residual Stresses and Cold Work," International Conference on the Fatigue of Metals, Institution of Mechanical Engrs., London (1956).

(22) L. P. Tarasov, W. S. Hyler, and H. R. Letner, "Effect of Grinding Conditions and Resultant Residual Stresses on the Fatigue Strength of Hardened Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 601 (1957).

(23) L. P. Tarasov and H. S. Grover, "Effects of Grinding and Other Finishing Processes on the Fatigue Strength of Hardened Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 668 (1950).

(24) M. F. Garwood, H. H. Zurburg, and M. A. Erickson, "Correlation of Laboratory Tests and Service Performance," Interpretations of Tests and Correlations with Service, Am. Soc. Metals (1951).

(25) W. S. Hyler, L. P. Tarasov, and R. J. Favor, "Distribution of Fatigue Failures in Flat Hardened Steel Test Bars," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 540 (1958).

(26) F. B. Stulen, H. N. Cummings, and W. C. Schulte, "Relation of Inclusions to the Fatigue Properties of High Strength Steels," International Conference on Fatigue of Metals, Institution of Mechanical Engrs., London (1956).

(27) W. L. Starkey, S. M. Marco, and R. R. Gatts, "Statistical Evaluation of Endurance Limit Among Several Heats of Propeller Type Steel," *TR 55-483* (*ASTIA No. AD 97190*), Wright Air Development Center, Aug. 1956.

(28) H. R. Bear and R. H. Butler, "Preliminary Metallographic Studies of Ball Fatigue Under Rolling Contact Conditions," *Technical Note TN 3925*, Nat. Advisory Committee for Aeronautics, March 1957.

APPENDIX I

X-RAY DIFFRACTION STUDIES OF SEVERAL 52100 STEELS AND TOOL STEELS

BY V. WEISS[8]

Finish-machined tension specimens from two heats of SAE 52100 steel and the three tool steels were subjected to an X-ray diffraction study in an attempt to determine residual stresses of first and second order as well as the presence or absence of retained austenite and carbides. These studies were made on a General Electric XRD-5 diffraction unit with a proportional counter using vanadium oxide filtered chromium Kα radiation.

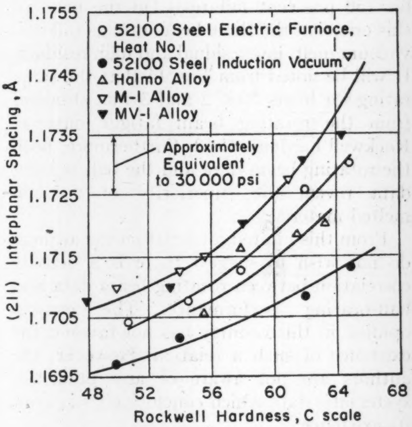


FIG. 20.—Interplanar Spacing of (211) Planes as a Function of Hardness for Various Bearing Steels.

The interplanar spacings of the (211) planes parallel to the surface are shown in Fig. 20 as a function of hardness and the (211) diffraction line width at one half intensity as a function of hardness in Fig. 21. Table VI gives the indications for retained austenite and carbides.

From Fig. 20 it can be seen that the *d*(211)-value increases for all steels with increasing hardness level. Since this *d*(211)-value was determined from planes oriented parallel to the specimen surface an increase in residual compressive stress is indicated. Based on this concept a change of the *d*(211)-value by 0.0010 Å represents a change in stress of approximately −30,000 psi. However, this figure can be used for order of magnitude type comparisons only.

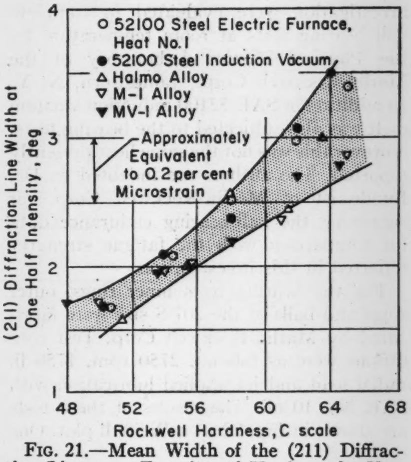


FIG. 21.—Mean Width of the (211) Diffraction Line as a Function of Hardness for Various Bearing Steels.

According to Fig. 21 the (211) line width also increases with increasing hardness level. Among the factors responsible for increase in diffraction line widths are microstrains, particle size, plastic deformation etc., the most significant contribution in the present case probably being microstrain. On this basis an increase of the line width at half intensity of 1 deg corresponds to an increase of the microstrain level of approximately 2×10^{-3}. This figure can also serve for semiquantitative evaluation only.

---

[8] Assistant Professor, Department of Chemical and Metallurgical Engineering, Syracuse University, Syracuse, N. Y.

The amount of retained austenite, Table VI, tended to increase somewhat at the higher hardness levels for all alloys. A similar increase was observed for the carbides except for the SAE 52100 vacuum melt and MV-1 alloy which gave no carbide indications at any hardness level. The differences between the various alloys in respect to retained austenite and carbides is considerable. Only traces were found for the two heats of SAE 52100 steel. In contrast, M-1 alloy showed strong indications of retained austenite and carbides at the highest hardness level (C 65). There does not, however, appear to be any definite correlation between these indications and the reported mechanical properties.

APPENDIX II

COMPARISON OF BALL-BEARING LIFE DATA WITH ROTATING-BEAM FATIGUE RESULTS

Two of the SAE 52100 steel electric furnace heats, Nos. 2 and 3 of the present investigation were evaluated in complete ball bearing tests at room temperature by the Physical Testing Laboratory of the Marlin-Rockwell Corp., Jamestown, N. Y. In addition, a SAE 52100 induction vacuum melt was also subjected to the bearing tests, however this was not the same heat presently reported. The authors are indebted to Dr. Lundquist of Marlin-Rockwell Corp. for supplying the ball-bearing endurance data for comparison with the fatigue strengths reported in this investigation.

For the bearing tests inner rings, outer rings and balls of the 207-S size were fabricated by Marlin-Rockwell Corp. Test conditions were as follows: 2750 rpm, 1750 lb radial load and jet applied lubrication with SAE No. 10 oil. The results of these tests are shown in Fig. 22 on a Weibull plot. One characteristic of bearing performance which can be derived from this plot is the median life (50 per cent failures). On the basis of this criterion the three heats rate as follows: vacuum melt, low residual and high residual. It will be noted from Fig. 14 that the same rating for heats Nos. 2 and 3 was obtained from the rotating beam fatigue data at Rockwell hardness C 62. Furthermore, both the rotating beam data and the ball-bearing data reveal the superiority of vacuum melted material.

From this limited information the authors do not wish to suggest there is a definite correlation between rotating beam data and ball-bearing performance. The general opinion in this country has not favored the existence of such a relation. However, the authors are not aware of any published systematic data which conclusively disprove its existence.

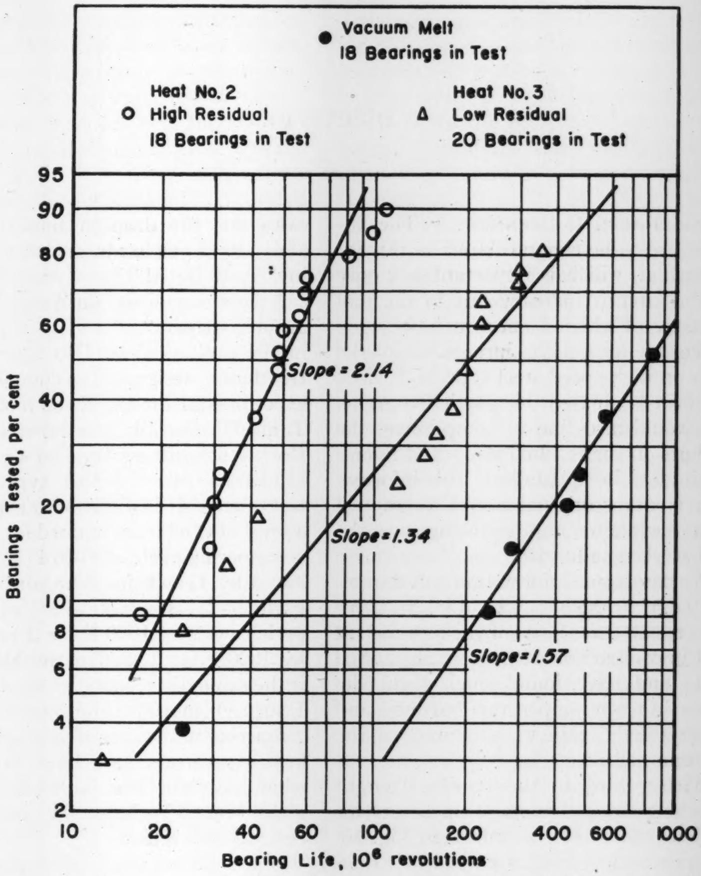


FIG. 22.—Complete Ball-Bearing Data for Various Heats of SAE 52100 Steel (M. R. C. Lundquist).

DISCUSSION

Mr. Ralph G. Kennedy.[1]—The authors are to be congratulated on this information, which is important not only to the aircraft industry but to the tool industry of which I am a member.

We are especially interested in the data on high-speed steel type M-1, since our firm originated this particular grade.

I would also like to compliment the authors on the residual stress and microstrain data in Appendix I. This information is quite original and I think will help to explain a number of things to the tool steel metallurgists.

We have some information on vacuum-melted M-1 which may be of interest. We have tested three heats of vacuum-melted M-1 in comparison with many air-melted heats and have found some slight decrease in room temperature hardness and a very slight gain in toughness in the vacuum-melted steels.

With regard to the tensile strength data measured at room temperature for various tool steels, as shown in Fig. 10, our experience with air-melted M-1 does not agree with the authors' for vacuum-melted M-1. At a hardness level of Rockwell C 65.5, we have observed a tensile strength for air-melted M-1 of about 383,000 psi.[2] We have found that the static room temperature properties of M-1 tend to increase with increasing Rockwell hardness up to at least a level of C 65.5. Could vacuum melting be the cause for the drop in tensile strength above Rockwell hardness C 62 as recorded in Fig. 10 for M-1?

I have a question on Table IV of the authors' paper.

The authors show two types of heat treatments designated as commercial and experimental for the three tool steels in Table IV. For all three types of steels a Rockwell hardness level of C 62 is obtained by two different types of heat treatments. It is difficult to explain how a level of C 62 was obtained for M-1 with a tempering cycle of 1000 F for 2 hr followed by 1120 F for 2 hr, and the same hardness level was obtained with a tempering cycle of 1000 F for 2 hr followed by 1000 F for 2 hr. For the MV-1 there is the same discrepancy at the C 62 level. I suspect that the time for the second tempering in the case of the experimental heat treatments may have been somewhat less than 2 hr, but I am not sure about that. Perhaps the authors can clear up this difficulty.

Since the authors have presented a great amount of data on yield strength and elastic limits, I believe it would be helpful to show some of the individual stress-strain curves from which the yield and elastic limits were selected so that we could better judge the value of the selection of such limits.

Mr. J. D. Marble.[3]—Our laboratory has done fatigue work on various bearing materials; some of the early work was reported by R. A. Baughman last fall before The American Society of Mechanical Engineers. We have developed a relatively simple apparatus in which contoured wheels bear upon the surface of the material. This subjects the material to compressive loading such as it actually experiences in a bearing application, rather than the tensile loading reported by the authors. There are a few other advantages which we could discuss, the most important being that the specimens are easy to prepare, are inexpensive, and furnish at least two tests per specimen.

---

[1] Director of Laboratories, The Cleveland Twist Drill Co., Cleveland, Ohio.

[2] R. G. Kennedy, "A Study of Sub-Zero Treatments Applied to Molybdenum-Tungsten High-Speed Steel," *Transactions*, Am. Soc. for Metals, Vol. 34, pp. 250–309 (1945).

[3] Specialist, Mechanics of Materials, Flight Propulsion Laboratory, General Electric Co., Cincinnati, Ohio.

Our findings do corroborate those of the authors, where they show that MV-1 steel increases its relative position at 500 F to the extent that it is equal to or better than the other alloys. In our investigation, MV-1 was somewhat stronger than Halmo and M-1 at these temperatures. Much of our investigation has been carried out at higher temperatures. Another investigation in this bearing material field which we should be reporting soon is the fatigue behavior of various bearing steels in the presence of some of the standard bearing lubricants.

MR. ROBERT A. HELLER.[4]—Why did the authors expect to find a relationship between the fatigue and static properties of these materials when it is well known that they are governed by different mechanisms and for no materials have such relationships been found?

MR. T. J. DOLAN.[5]—In Fig. 14 of the paper the data indicate that the induction-vacuum-melted steel had a higher endurance limit than the electric furnace melt throughout the range of hardness investigated. However, if one analyzes the data in Fig. 13, it is evident that the induction-vacuum-melted steel had a great deal more scatter in fatigue life than the electric furnace steel. This would indicate that on the basis of life to failure in a bearing the induction-vacuum-melted steel would be a much poorer bearing material. These inconsistencies in life indicate the presence of erratic abnormal conditions that would lead to premature failure in an occasional bearing element.

It would be helpful to know whether the authors have similar data to that in Fig. 13 for the other hardness levels and whether they show this same greater amount of scatter for induction melted steel as compared to the electric furnace steel.

One other point might be clarified in connection with the preparation of specimens. The paper indicates the samples were finished 0.035 in. over-sized, heat-treated, then finish-machined by grinding and polishing. Since the fatigue characteristics in small specimens are vastly affected by the method of processing the final surfaces, the method of finishing the surface might be a factor in affecting the differences observed between some of the steels or heat treatments involved in the tests.

MR. R. A. LULA.[6]—I would like to ask the authors whether they have any information on consumable electrode-melted material as compared to induction vacuum.

MR. MARBLE.—There is one little additional point we should mention. Surface finish becomes much less critical in a testing procedure where the surface is loaded in compression and the failure is by subsurface shear. Thereby most of the variability introduced by the specimen preparation procedure is eliminated. We have found this to be an advantage in our testing.

There was one question about the relative strength of the two vacuum-melting methods—induction *versus* consutrode. For more details, you could contact our group, but our finding is that the consutrode vacuum-melted material has been definitely superior.

---

[4] Assistant Professor, Columbia University, New York, N. Y.

[5] Head, Department of Theoretical and Applied Mechanics, Talbot Laboratory, University of Illinois, Urbana, Ill.

[6] Chief Research Metallurgist, Allegheny Ludlum Steel Corporation, Tarentum, Pa.

MESSRS. G. SACHS, R. SELL, AND W. F. BROWN, JR. (*authors' closure*).—The authors wish to thank those who contributed to the discussion.

Mr. Kennedy raises some questions concerning the influence of vacuum melting on the room temperature tensile properties of tool steels. The authors know of no reason why vacuum melting *per se* should cause a decrease in tensile strength at high hardness levels. The decrease in tensile strength shown in Fig. 10 may be associated with residual stresses as pointed out in the paper. The magnitude of these stresses will vary with the geometry of the quenched blank, the heat treatment and to some extent with composition. It is to be noted that air-quenched Halmo, Fig. 10, does not show a drop in tensile strength at the highest hardness. This may be explained by the lower residual stresses associated with air cooling compared with oil quenching. Mr. Kennedy also raises a question regarding the hardness values reported in Table IV for the experimental and commercial heat treatment. The authors have re-examined the heat treatment information furnished by Marlin Rockwell Corp. The tempering procedure for the experimental heat treatment was in some instances varied from that given in Table IV. These variations were either in the direction of increasing the time or slightly raising the temperature in order to meet the specified hardness requirements. Such variations should not influence the results when data are compared on the basis of equivalent hardness, as is done in this paper. Regarding Mr. Kennedy's comment concerning the yield strength and elastic limits, the authors have stated in the paper that no quantitative elastic limits could be determined by the techniques employed. However, it is also pointed out that the yield strength values were determined using both with wire resistance gages and extensometers. The authors believe these values to be accurate.

Mr. Marble indicates a simulated bearing test procedure shows a relative improvement in the performance of MV-1 at 500 F. Unfortunately, the authors fatigue data do not clearly define differences in the fatigue characteristics of the tool steels at either room temperature or 500 F.

The comment by Mr. Heller neglects already established facts. Thus, in general, the fatigue strength of heat-treated steels increases with the tensile strength over a rather wide range of tensile strength values. Furthermore, the bearing life is known to increase with the hardness up to very high hardnesses. In these cases, the failure mechanisms are apparently different. However, certain common features exist which give rise to the relations. Thus, static tensile properties are primarily dependent on resistance to plastic flow. In fatigue, fracture is preceded by plastic flow but on a very small scale. Increasing the resistance to plastic flow would therefore be expected to increase the fatigue life. This behavior is followed until other effects such as high residual stresses enter the picture.

The authors agree with Mr. Dolan's observation that scatter appears to be greater in the case of vacuum melted SAE 52100 than in the case of air melted material. The authors have no data other than that given in Figs. 12 and 13 and can give no reason for these differences in scatter. However, there does seem to be general agreement in the bearing industry that vacuum melting improves bearing performance. The authors are well aware of the influence of surface finish on the fatigue life of small specimens. In the present investigation, care was taken to prepare all specimens in the same manner. Therefore, it is not believed that differences in surface finish contribute to the differences in fatigue life.

Regarding the question by Mr. Lula, the authors have no information directly relating to the relative effects of induction vacuum *versus* consumable electrode vacuum melting.

The authors wish to call attention to certain statements made in the paper concerning the possible influence of residual elements on both the fatigue life and bearing performance of SAE 52100. It has been brought to their attention that the copper, nickel, and molybdenum contents of the various heats of SAE 52100 are well within the limits specified for this steel by both the AISI and ASTM. The suggestion has been made that the differences in mechanical properties may be explained by variations in inclusion content and not by residual element differences. Examination of the inclusion data in Table II does not in itself lend support to this hypothesis. However, the authors do not deny the possibility and have emphasized in the paper the difficulty of correlating fatigue life with inclusion content. The authors are not aware of any systematic investigations dealing with the influence of residual elements. Such investigations are necessary before definite conclusions regarding the effects of these elements can be established.

In conclusion, it should be pointed out that the large volume of fatigue test data available for 4340 and 52100 steels varies to a much greater extent than can be explained by differences in testing procedures. Apparently, no systematic study of the sources of these discrepancies has been yet attempted. It may be suspected that either composition or processing techniques[7] affect considerably the endurance limit of heat treated steels. Regarding vacuum melting, for example, it appears established that it improves minimum stress values at the endurance limit. However considerable overlap exists between the data reported for air and vacuum melted steel.

---

[7] For example, repeated (double) tempering has been found to increase considerably the fatigue strength of modified 4335 steel.

THE EFFECT OF STRESS CYCLING ON THE STATIC MECHANICAL PROPERTIES OF SAE 4340 STEEL*

By J. Marin,[1] P. Borachia,[1] and U. A. Rimrott[1]

Synopsis

The main objective of this study is to investigate the influence of precycling on the various static tensile properties of the material. To the author's knowledge, previous investigations have been confined to the effect of precycling on the dynamic properties and not the static properties. Specimens of SAE 4340 heat-treated steel were precycled under axial tensile loading to various percentages of the fatigue life for the stress level selected. From the stress-strain curves obtained, the various tensile mechanical properties were determined and compared with the property values for specimens not subjected to initial precycling. As a result of the foregoing tests it was found that precycling produced small changes in the properties: it reduced the hardness and ductility but increased the ultimate strength and strain hardening exponent.

Various theories have been proposed to explain the mechanism of fatigue. Some of these theories are based upon the concept that materials have a certain available ductility and that under the condition of stress cycling, failure will occur when the resultant permanent strain or ductility has reached the fracture strain under static loading. This concept of "depletion of ductility" as the basis for fatigue failure was approximately supported in preliminary studies by Sachs (**1**)[2]. Another objective of this paper is to study this hypothesis. The results of the investigation do not support the suggested "depletion of ductility" hypothesis.

Material and Specimens

The material tested was an SAE 4340 steel, heat treated by quenching and tempering at about 1250 F. This treatment resulted in a steel having a nominal ultimate strength of about 140,000 psi and a yield strength of about 128,000 psi. The alloy had the following chemical composition, in per cent: carbon, 0.395; manganese, 0.76; silicon, 0.31; nickel, 1.78; chromium, 0.84; molybdenum, 0.23; sulfur, 0.016; and phosphorus, 0.008.

The specimen used had a constant circular cross-section 0.25-in. in diameter for a length of 1 in. as shown in Fig. 1. The specimen ends were designed so that the specimen could be used in both static and fatigue testing machines. The specimen length was selected to prevent buckling during the compression stage of

---

* Presented at the Sixty-second Annual Meeting of the Society June 21–26, 1959.

[1] Department of Engineering Mechanics, The Pennsylvania State University, University Park, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

the stress cycle. The gage length section of the specimens was polished to a 2 microinch rms finish.

TESTING EQUIPMENT

Equipment for Static Tests:

A Baldwin Universal testing machine was used for the static tests. To obtain an accurate evaluation of the effect of stress cycling upon the static tensile mechanical properties it is necessary to obtain the true stress-strain diagrams. For this purpose the axial strains were measured by the specially designed direct measuring instrument shown in Fig. 2. By means of the vertically adjustable telescope and dial gage shown, the axial elongation can be determined for a selected gage length. For each load increment, the diameters of the specimens were obtained directly by use of a micrometer since beyond the point of instability or maximum load, the localized strain is more accurately evaluated by the diameter changes than by the axial strains. Prior to testing, the load indicator on the machine was calibrated and appropriate corrections to the load readings were made.

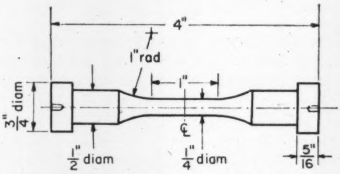


FIG. 1.—Fatigue Specimen.

Equipment for Fatigue Tests:

Axial fatigue tests were made on an SF-1-U Sonntag fatigue testing machine. This machine was equipped with a magnifying lever having a 5:1 ratio so that a completely reversed load up to ±5000 lb could be axially applied to the specimen. By means of two preload helical springs, a static mean axial tensile load up to 5000 lb could be superimposed upon the completely reversed load. Because of the type of shank on the tension specimens, special spherical grips were required for attaching the specimens in the fatigue testing machine (Fig. 3). By use of these grips, eccentricity of loading and bending stresses were eliminated. Preliminary fatigue tests showed that there was no measurable relative motion between the grips and the specimen. It was also demonstrated that any

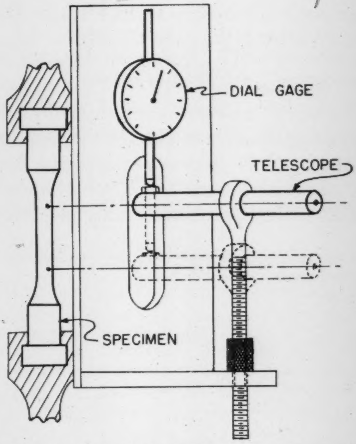


FIG. 2.—Principle of Device for Measuring the Elongation of the Specimen in the Static Tension Testing Machine.

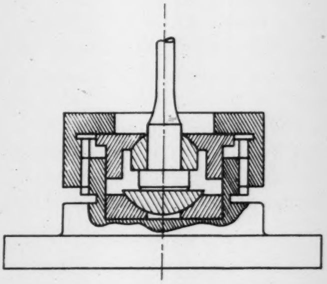


FIG. 3.—Spherical Grip.

impact forces produced as a result of load reversals were of negligible magnitudes. Initially, hysteresis loops were obtained in order to determine if a change in hysteresis occurs with stress cycling. Unfortunately, the results could not be used since the hysteresis loss in the SR4-gages varied from gage to gage.

Equipment for Hardness Tests:

Hardness values were taken for each specimen in order to correct for variation in the material between specimens. During the initial tests, the Knoop hardness values were measured with a Tukon hardness tester. Later a Durimet Leitz microhardness tester was used for this purpose so that hardness values could be obtained using very small indentations. With the microhardness tester, it was possible to obtain hardness readings on the polished gage length section of the specimens without damaging the specimens.

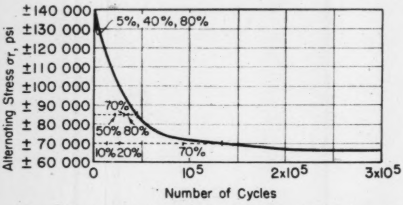


FIG. 4.—Percentage of Precycling for Stresses Completely Reversed. Percentages denote percentage of life precycled at stress values noted.

TEST RESULTS AND THEIR INTERPRETATION

Fatigue Test Results:

To determine the influence of precycling upon the mechanical properties of materials, it was first necessary to establish the standard σ-N diagrams for the material. For this purpose, fatigue strength values were obtained for completely reversed stresses at three stress levels and for four stress levels in which the stresses varied from zero to a maximum value.

The completely reversed stresses were conducted for two stress levels below the static yield stress and one stress level above the yield stress (Fig. 4). For the case in which the stresses varied from zero to a maximum, the four stress levels were all below the yield stress with one stress level being below the endurance limit (Fig. 5). The specimens used for each stress level were selected by sorting the specimens according to hardness values. In this way, the scatter of data was reduced. The results of the fatigue tests are shown in Fig. 6.

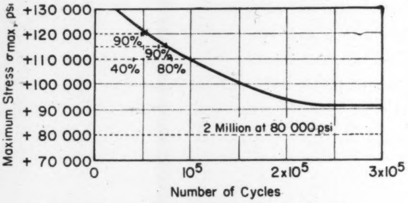


FIG. 5.—Percentage of Precycling for Stresses Varying from 0 to Maximum. Percentages denote percentage of life precycled at stress values noted.

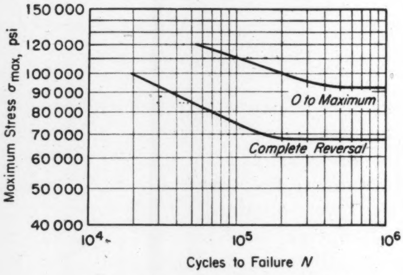


FIG. 6.—σ-N Diagrams.

To determine influence of precycling on the static mechanical properties, groups of specimens were precycled at the various stress levels for selected percentages of the average fatigue life at the selected stress levels. The various percentages of precycling used and corresponding stress cycles are shown in Figs. 4 and 5.

In selecting the type of stress cycling and the magnitudes of the maximum stresses, an attempt was made to cover the range of values as completely as possible.

Hardness Test Results:

Knoop hardness values were initially obtained using a Tukon hardness tester for all 200 specimens used in this investigation. The readings were taken on the cylindrical surface of the specimen ends. Six readings were obtained alternately for each end of each specimen and the data were analyzed statistically. By examining the variance of the data, it was found that the variance between specimens was 2.671 times the variance of the residual error. The maximum theoretical value of the variance at 5 per cent probability (for the case of no difference between specimens) was found to be only 1.08 times the variance of the residual error. An analysis of the data also showed that no significant change in the testing apparatus occurred during the time of the tests. The foregoing analysis of the hardness data indicates that the variation of the hardness between specimens is much greater than the variation between readings for an individual specimen. The static properties that would be obtained with no stress cycling were determined by assuming a linear relation between Knoop hardness and other static properties, using the test results for specimens that were not precycled. These values are listed in Tables I and II.

TABLE I.—STATIC NOMINAL PROPERTIES WITHOUT PRECYCLING.

Specimen	Knoop Hardness	Vickers Hardness at the Ends of the Specimens	Vickers Hardness at the Gage Length of the Specimens	Difference in Hardness, per cent	Yield Strength, 1000 psi	Ultimate Strength, 1000 psi	Elongation, per cent	Reduction of Area, per cent	Toughness Index Number, 1000 psi
No. 133......	333.72	335.6	328.9	−2.0	125.8	138.0	25.4	61.07	35.1
No. 178......	338.72	337.8	334.1	−1.1	128.0	141.0	25.5	58.5	36.0
No. 169......	343.84	339.8	335.7	−1.3	130.8	144.3	25.3	60.5	36.5
Average Values...	338.76	337.8	332.9	−1.5	128.2	141.1	25.4	60.0	35.8

TABLE II.—STATIC TRUE PROPERTIES WITHOUT PRECYCLING.

Specimen	Ultimate Strength, 1000 psi	Ductility per cent Strain[a]	Ductility per cent Strain[b]	Toughness, 1000 psi	Strain-hardening, exponent *n*
No. 133...........	150.2	8.9	94.3	12.4	0.130
No. 178...........	154.7	9.7	87.5	13.8	0.132
No. 169...........	156.5	9.0	92.7	12.8	0.144
Average values..	153.8	9.2	91.5	13.0	0.135

[a] Based upon true strain at ultimate load using diameter readings.

[b] Based upon true strain at fracture load using diameter readings.

To determine the influence of precycling on hardness, Vickers hardness values were taken by means of a Durimet Leitz microhardness tester after precycling and before the static testing. Ten indentations were made over the gage length and along a straight line with a distance of 2 mm between the microhardness indentations. The outer indentations were later used as marks for measuring the axial strains. To correlate the hardness after cycling with the values before cycling, Vickers microhardness values were also obtained on the cylindrical ends of the cycled specimens. Since it can be assumed that the specimen ends were not influenced by precycling, the change in hardness due to precycling could in this way be determined. A small correction of 1.5 per cent was made in the foregoing data to provide for the difference between the hardness of the specimen ends and the hardness of the gage length. This difference in hardness may have been due to variations in heat and surface treatments.

TABLE III.—SUMMARY OF CHANGES IN NOMINAL MECHANICAL PROPERTIES.

Stress Variations, psi	Percentage of Life Precycled	Average Change of Property, per cent					
		Vickers Hardness	Yield Strength	Ultimate Strength	Elongation	Reduction of Area	Toughness Index Number
−133 000 to +133 000..	5	−1.0	−3.2	−2.4	−1.5	−1.3	−3.5
	40	−2.9	−1.6	+2.4	−2.5	−4.9	−0.2
	80	−1.6	−9.2	−5.3	+4.6	−0.6	−1.1
−85 000 to +85 000....	50	−5.5	+0.6	+1.7	−2.5	−1.5	−0.8
	70	−2.7	−1.9	+0.7	+0.8	−1.2	+1.5
	80	−2.5	+1.4	+1.3	0	−1.1	+1.3
−70 000 to +70 000....	1	−2.3	+1.6	−0.1	+0.2	+0.2	−0.3
	20	−0.6	+1.7	+0.5	+2.0	−1.2	+3.0
	70	+1.4	+1.2	+0.8	+1.5	+0.5	+2.4
0 to 120 000............	90	−2.0	+2.5	+3.4	+0.4	−1.3	+3.6
0 to 115 000............	90	−1.8	+1.9	+1.9	−4.2	0	−1.2
0 to 110 000............	40	−2.5	+2.6	+2.0	+4.7	+0.2	+7.3
	80	−1.4	+0.9	+1.1	+4.7	−1.2	−3.7
0 to 80 000.............	2 000 000 cycles	−1.1	+3.7	+2.1	+2.9	+1.4	+6.7
Average, per cent change		−1.9	+0.2	+0.7	+0.8	−0.9	+1.1

NOTE.—Specimens with definite irregular behavior were omitted in determining the values in the above table.

The average values of the initial hardness and change in hardness due to precycling were considered in the property values reported in Table III.

Static Tension Test Results:

Control specimens were tested in static tension. The average nominal and true stress-strain diagrams for these tests are shown by the solid lines in Figs. 7 to 12. These average relations are based on the values for the specimens shown in Tables I and II with a correction for the variation in hardness as described above.

The nominal mechanical properties were determined based upon the following:

Yield Strength as measured by the yield stress or:

$$\sigma'_y = \frac{P_y}{A_0} \quad \text{..............} \quad (1)$$

Nominal Ultimate Strength as measured by the nominal ultimate stress or:

$$\sigma'_u = \frac{P_u}{A_0} \quad \text{..............} \quad (2)$$

Nominal Ductility based upon the

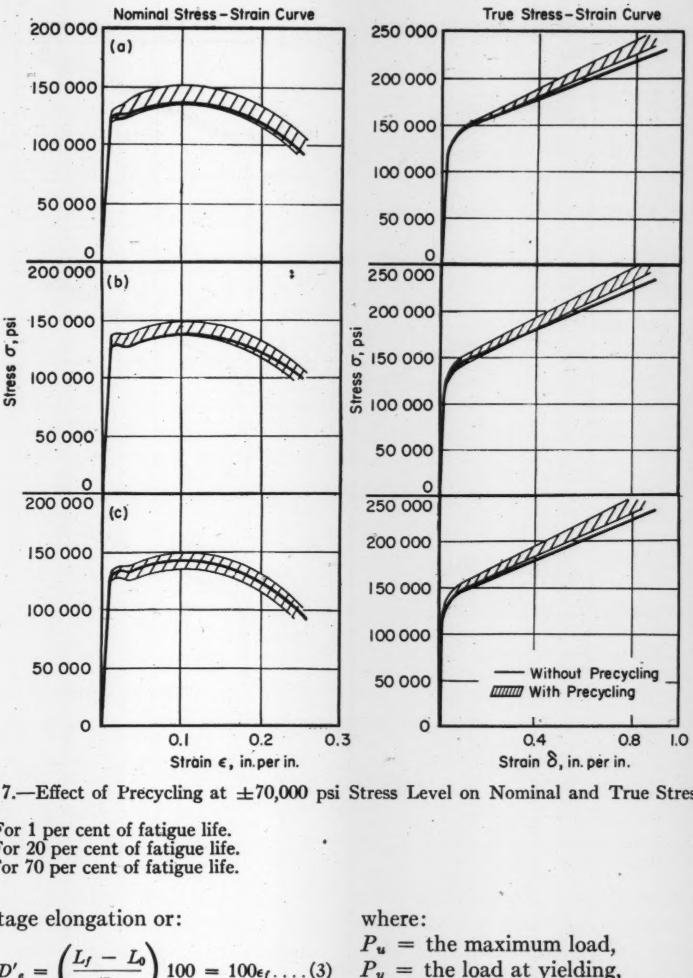


FIG. 7.—Effect of Precycling at ±70,000 psi Stress Level on Nominal and True Stress-Strain Curves.
(*a*) For 1 per cent of fatigue life.
(*b*) For 20 per cent of fatigue life.
(*c*) For 70 per cent of fatigue life.

percentage elongation or:

$$D'_e = \left(\frac{L_f - L_0}{L_0}\right) 100 = 100\epsilon_f \ldots . (3)$$

Nominal Ductility based upon the percentage reduction of area or:

$$D'_a = \left(\frac{A_0 - A_f}{A_0}\right) 100 \ldots\ldots . (4)$$

Nominal Toughness as measured by the toughness index number or:

$$T' = \sigma'_u \cdot \epsilon_f \ldots\ldots\ldots\ldots (5)$$

where:
P_u = the maximum load,
P_y = the load at yielding,
A_0 = the original cross-sectional area,
A_f = the cross-sectional area at fracture,
L_0 = the original gage length,
L_f = the gage length at fracture, and
ϵ_f = the nominal strain at fracture.

Since the mechanical properties based upon the true stress and strain give a more accurate determination of the static properties, their values were also determined. Figures 7 to 12 show the

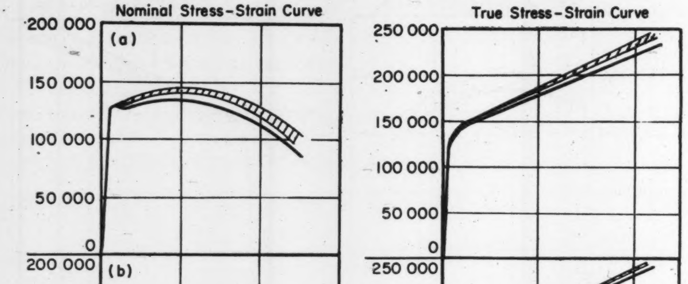


FIG. 8.—Effect of Precycling at ±85,000 psi Stress Level on Nominal and True Stress-Strain Curves.
(*a*) For 50 per cent of fatigue life.
(*b*) For 70 per cent of fatigue life.
(*c*) For 80 per cent of fatigue life.

true stress-strain relations based upon the actual cross-sectional area A rather than the original cross-sectional area A_0. That is, the true stress and true strain are equal to:

$$\sigma = \frac{P}{A} = \frac{4P}{\pi d^2} \quad \text{(6)}$$

$$\delta = \int_{L_0}^{L} \frac{dL}{L} = \log_e \frac{L}{L_0} = \log_e \left(\frac{d_0}{d}\right)^2 \quad \text{(7)}$$

where:

d = the actual diameter of the specimen of the load P, and

d_0 = the original diameter.

Equation 7 is based upon the condition that the volume of the material remains constant in the plastic range. Based upon the definitions of true stress and strain, as given by Eqs 6 and 7, the following true mechanical properties can be defined:

True Ultimate Strength as determined by the true stress at the ultimate load or by Eq 6:

$$\sigma = \frac{P_u}{A_u} = \frac{4P_u}{\pi d_u^2} \quad \text{(8)}$$

True Ductility as measured by the

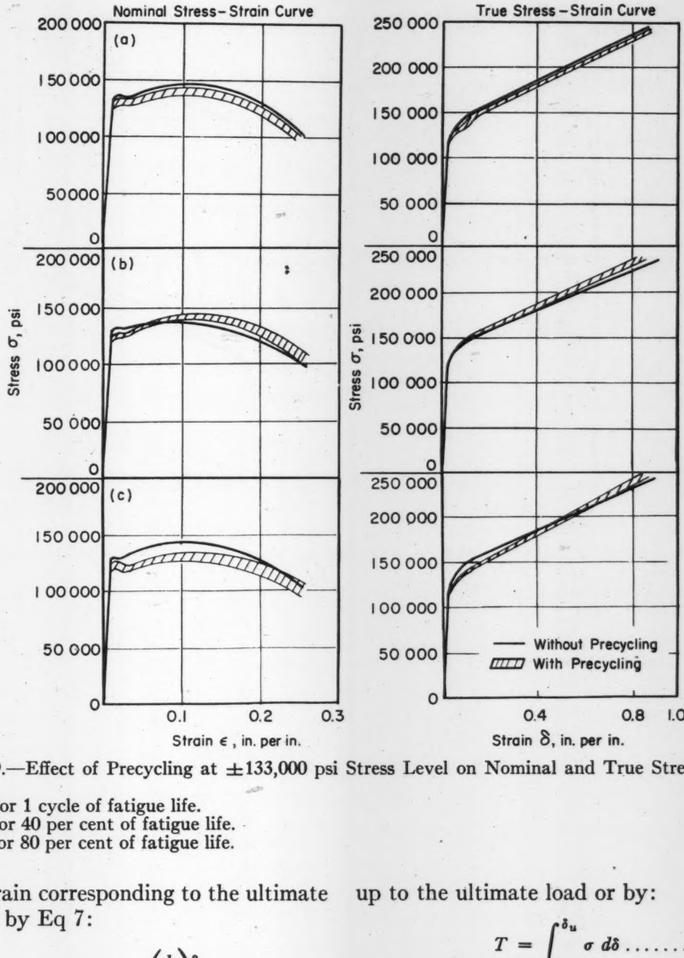


Fig. 9.—Effect of Precycling at ±133,000 psi Stress Level on Nominal and True Stress-Strain Curves.

(*a*) For 1 cycle of fatigue life.
(*b*) For 40 per cent of fatigue life.
(*c*) For 80 per cent of fatigue life.

true strain corresponding to the ultimate load or by Eq 7:

$$\delta_u = \log_e \left(\frac{d_0}{d_u}\right)^2 \quad (9)$$

The true strain at fracture was also determined as a measure of ductility. However, the combined stresses provided in the necked region of the specimen make this latter measure of ductility inexact.

True Toughness as measured by the area under the true stress-strain diagram up to the ultimate load or by:

$$T = \int_0^{\delta_u} \sigma \, d\delta \quad (10)$$

The property of strain hardening can be obtained by assuming that the true stress-strain data can be represented by a straight-line relation on a log-log plot. That is,

$$\sigma = k\delta^n \quad (11)$$

where:

k = the strength coefficient, and
n = the strain hardening exponent.

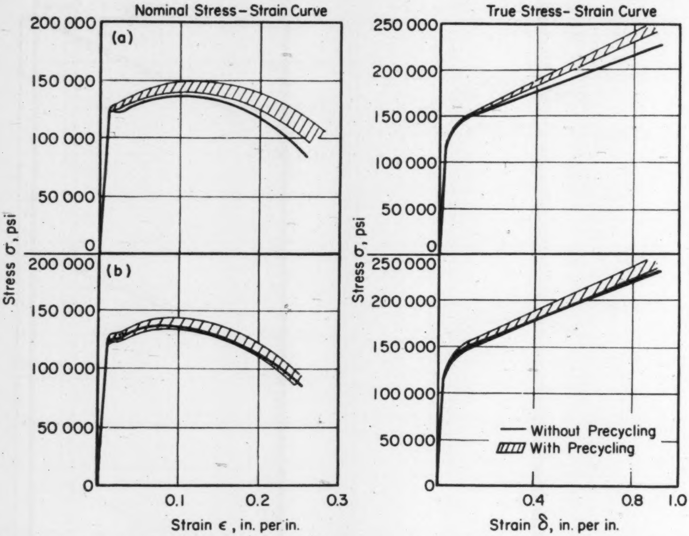


FIG. 10.—Effect of Precycling at 0 to +110,000 psi Stress Level on Nominal and True Stress-Strain Curves.
(*a*) For 40 per cent of fatigue life.
(*b*) For 80 per cent of fatigue life.

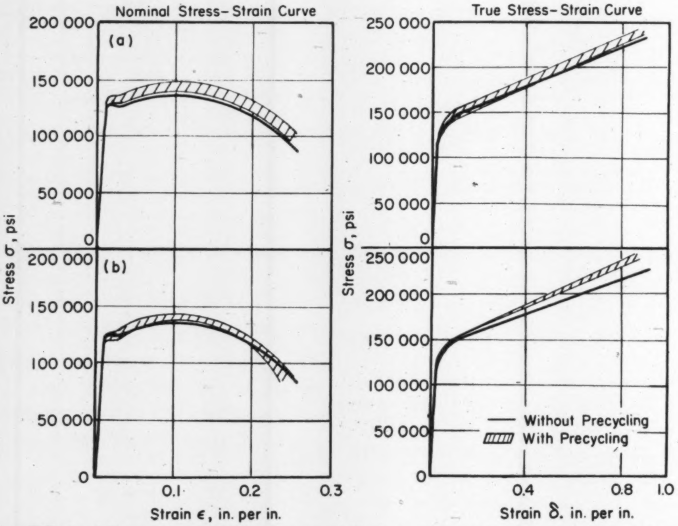


FIG. 11.—Effect of Precycling for 90 per cent of Fatigue Life on Nominal and True Stress-Strain Curves.
(*a*) At 0 to 120,000 psi stress level.
(*b*) At 0 to 115,000 psi stress level.

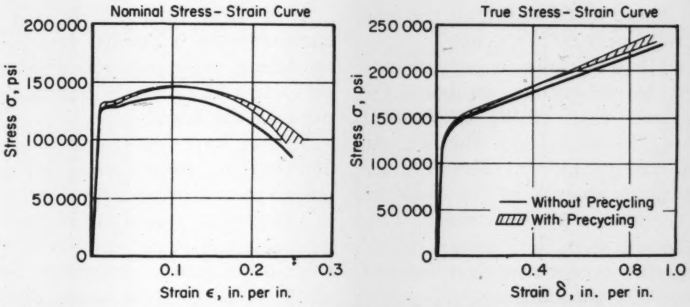


FIG. 12.—Effect of Precycling for Two Million Cycles at 0 to +80,000 psi Stress Level on Nominal and True Stress-Strain Curves.

TABLE IV.—SUMMARY OF CHANGES IN THE TRUE MECHANICAL PROPERTIES.

Stress Variation, psi	Percentage of Life Precycled	Average Change of Property, per cent				
		Ultimate Strength	Ductility per cent Strain[a]	Ductility per cent Strain[b]	Toughness	Strain-hardening, exponent *n*
−133 000 to +133 000...	5	−1.7	−1.7	−1.9	−7.0	+18.5
	40	+1.9	+3.5	−7.6	+3.8	+12.5
	80	−2.7	+30.2	−0.6	+13.3	+16.2
−85 000 to +85 000.....	50	+4.3	+26.7	−2.0	+12.0	+9.8
	70	+2.9	+6.4	−1.6	+16.5	+10.0
	80	+2.8	+6.7	−1.6	+3.2	+14.9
−70 000 to +70 000.....	20	+2.5	+13.1	−4.0	+4.8	0
	70	+3.7	+16.2	−1.1	+8.4	+14.1
0 to 120 000............	90	+4.5	+4.8	−2.6	0	+23.0
0 to 115 000............	90	+7.7	+34.1	+0.8	+38.0	+22.8
0 to 110 000............	40	+2.3	−2.2	+0.7	+5.0	+18.5
	80	+1.6	−8.5	−2.3	−4.1	+3.4
0 to 80 000.............	2 000 000 cycles	+3.1	+16.3	+2.8	+11.7	+14.3
Average, per cent change..........		+2.5	+11.2	−1.6	+8.1	+13.7

[a] Based upon true strain and ultimate load using diameter readings.

[b] Based upon true strain at fracture load using diameter readings.

NOTE.—Specimens with definite irregular behavior were omitted in determining the values in the above table.

The values of the strain hardening exponent *n* can be obtained by plotting the test data on log-log paper. Then the slope of the straight line (Eq 11) representing the data is equal to *n*.

Figures 7 to 12 show the nominal and true stress-strain curves for the various specimens subjected to different amounts of precycling, as compared to the average stress-strain curves for the material without precycling. An examination of these diagrams shows that, in practically all cases, there was a raising of the true stress-strain curve by precycling.

Tables III to IV summarize the change produced by precycling on both the nominal and true tensile mechanical properties. To determine whether the property changes due to precycling are significant, an analysis of the variance (10) of the test results was made. This study showed that the changes in all the properties except true ductility and true toughness are significant.

CONCLUSIONS

The test results for the heat-treated SAE 4340 steel given in Tables III and IV together with the statistical analysis shows that:

1. For all prestress conditions, except for 70 per cent precycling at ±70,000 psi, precycling slightly reduces the hardness.
2. For most prestress conditions, precycling slightly increases both the true and nominal ultimate strength.
3. For most prestress conditions, precycling slightly decreases the nominal ductility as determined by the reduction in area.
4. A large increase in the strain hardening (as measured by the strain hardening exponent) was produced by precycling. An average increase of 13.7 per cent was found.
5. The hypothesis that ductility is depleted by stress-cycling is not supported by this investigation.

The general conclusion is that for the material and stress conditions used in this investigation precycling has only small effects on the static mechanical properties.

Acknowledgments:

The sponsorship and financial assistance of the Wright Air Development Center in making possible this investigation and the technical assistance of W. J. Trapp and C. L. Harmsworth are greatly appreciated. The authors also appreciate the assistance of C. L. Harmsworth in testing the fatigue specimens at ±85,000 and ±133,000 psi. The technical advice of G. U. Oppel of the Department of Engineering Mechanics of The Pennsylvania State University is also appreciated.

REFERENCES

(1) George Sachs, "Effect of Strain at Fracture," *Fracturing of Metals*, Am. Soc. Metals, pp. 51–67 (1948).

(2) H. N. Cummings, F. B. Stulen, and W. C. Schulte, "Investigations of Materials Fatigue Problems Applicable to Propeller Design," *Technical Report 54-531*, Wright Air Development Center, May 1955.

(3) R. B. Davies, "Hardness Changes During Fatigue Tests on Copper," International Conference on Fatigue of Metals, London (England), Session 6, Paper 9, The Institution of Mechanical Engineers, London (England) (1956).

(4) A. M. Freudenthal and J. H. Weiner, "On the Thermal Aspect of Fatigue," *Journal of Applied Physics*, Vol. 27, Jan. 1956, pp. 44–50.

(5) H. J. Gough, and W. A. Wood, "Deformation and Fracture of Mild Steel under Cyclic Stresses in Relation to Crystalline Structure," *Proceedings*, Institution Mechanical Engrs. (London), Vol. 141, p. 175 (1939).

(6) John Kenyon, "The Reverting of Hard-Drawn Copper to Soft Condition under Variable Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 1073 (1950).

(7) J. Marin, "Engineering Materials," Prentice-Hall Inc., New York, N. Y., pp. 43–60, 178–221 (1952).

(8) W. L. Starkey, S. T. Marco, and R. R. Gatts, "Determination of the Effect of Fatigue Cracks on the Fatigue Properties of Metals," Report by the Ohio State University for Wright Air Development

Center, The Ohio State University Research Foundation, July 1957.

(9) Takeo Yokobon, "Damage as an Initial Stage of Fatigue-Fracture," *Journal*, the Physical Soc. of Japan, Vol. 8, p. 806 (1953).

(10) R. W. Guard, "Multi-Factorial Experiments in Materials Development," *Proceedings Statistical Methods in Materials Research*, Pennsylvania State University, Department of Engineering Mechanics, pp. 23–31 (1956).

DISCUSSION

MR. C. E. FELTNER[1] (*presented in written form*).—This paper was of great interest since similar static tension data for nearly the same material and heat treatment has been obtained in a recent investigation (**1**)[2] at the University of Illinois.

The material used was SAE 4340 aircraft quality steel, heat-treated by quenching and tempering at 1200 F. This treatment resulted in a steel having an average Knoop hardness of 312, a nominal ultimate strength of 138,700 psi, and a nominal 0.2 per cent offset yield strength of 127,200 psi. The alloy steel had the following chemical composition, in per cent: carbon, 0.40; manganese, 0.81; silicon, 0.25; nickel, 1.73; chromium, 0.87; molybdenum, 0.24; sulfur, 0.019; and phosphorus, 0.018. This alloy and treatment compare closely with that used by the authors.

A solid cylindrical static tension specimen 0.150 in. in diameter for a length of 1 in. was used. An Instron testing machine with a crosshead speed of 0.02 in. per min was used to apply and record the load. A modified version of a double ring diameter gage as described by Pian and D'Amato (**2**) was used to measure the decrease in diameter of the specimen. The load and actual diameter measurements are the only data necessary to construct the true stress-strain curve.

The average true stress-strain diagram obtained in this investigation is shown in the accompanying Fig. 13.

In their tests, the authors state that the load was applied in increments and that the diameter was measured with a micrometer beyond the point of maximum load, the apparatus in Fig. 2 being used for strain measurements prior to the maximum load. It seems that some difficulty might be encountered in making strain measurements by this method for the region above the yield point (for this particular material and heat treatment there is a well defined yield point). The reason for this is that for most materials a large amount of load relaxation and an increase in strain occurs in this region when the testing machine is stopped. However, the authors' results in Figs. 7 through 12 of their paper compare closely with those obtained at the University of Illinois as shown by Fig. 13.

In Eq 11, δ is defined by Eq 7 which is the equation for the *total* true strain. Another manner in which Eq 11 may be written (**3**) is,

$$\sigma = K(\delta_p)^n \ldots\ldots\ldots\ldots (12)$$

where δ_p is the true plastic strain and is equal to the total true strain minus the elastic strain or in equation form,

$$\delta_p = \left[\log_e \left(\frac{d_0}{d}\right)^2\right] - \frac{\sigma}{E} \ldots\ldots (13)$$

where E is the modulus of elasticity and the other terms are the same as those defined by the authors. Another way in which the true plastic strain may be defined is,

---

[1] Graduate Fellow, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this discussion.

$$\delta_p = \log_e \left[\left(\frac{d_0}{d} \right)^2 - \frac{\sigma}{E} \right] \quad \ldots\ldots \quad (14)$$

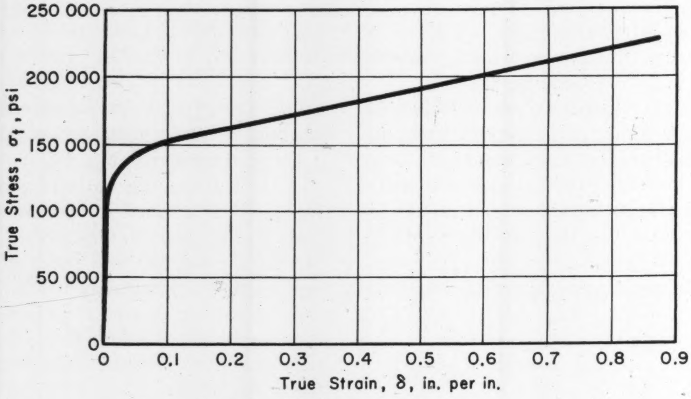


FIG. 13.—True Stress-Strain Curve.

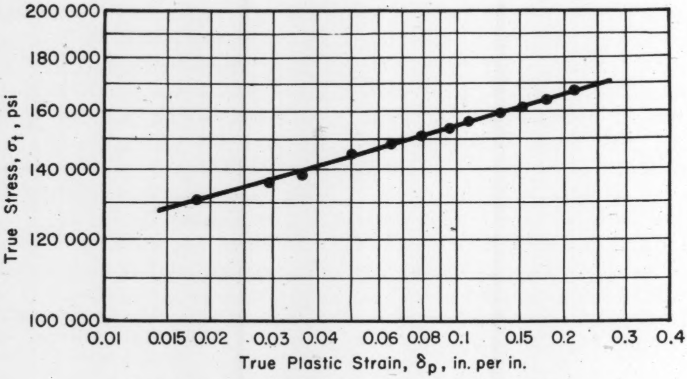


FIG. 14.—Logarithmic Stress - Plastic Strain Curve.

The percentage difference between Eqs 13 and 14 is only about 0.5 per cent for values of strain up to the point of maximum load, the variation becoming greater as the strain increases. Equation 13 seems to define more logically the true plastic strain than does Eq 14. Since strain hardening is usually associated with plastic strain, it seems logical to use only the plastic strain in the equation employed to calculate the strain hardening exponent. The fact still remains that Eq 12 and Eq 11 of the authors' paper are arrived at empirically from plots of the experimental data.

For this material the strain hardening exponent is determined in the region where the total true strain ranges from zero to about 0.10 in. per in. (approximately the point of maximum load). In the region slightly above the yield point the elastic strain may be as much as 50 per cent of the total strain. As the total strain increases, the elastic strain gradually becomes a smaller percentage of the total strain. For this reason the use of the total strain in Eq 11 will give strain hardening exponents of higher value than when the true plastic strain is used. A synthesis of a large amount of true stress-strain information (4) has shown that Eq 12 of this discussion fits the data of several materials very well.

In the work at Illinois on the SAE 4340 steel previously mentioned, the data fitted a straight line on a log-log plot better when the true plastic strain was used. These results are shown in Fig. 14. The average strain-hardening exponent is $n = 0.0985$, whereas the authors average value is $n = 0.135$. The difference is due in part to the different definitions of δ employed.

Using the average values of the properties given in Table II of the authors' work, it is possible to calculate the true stress for particular values of total true strain by means of Eq 11. The elastic strain can then be computed for the calculated values of true stress and subtracted from the total true strain leaving only the true plastic strain. By calculating the true stress and true plastic strain at two different points, the new strain hardening exponent, based on true plastic strain, may then be computed by means of Eq 12. The results of these calculations give a value of $n = 0.123$. The variation between the two different strain hardening exponents is about 10 per cent which may be a significant amount in some applications. In the authors' work, this variation may not be of too much importance since the true stress-strain curve was used only on a comparative basis. It was of particular purport to me because of the manner in which the strain-hardening exponent is used in reference (1).

MR. JOSEPH MARIN (*author*).—The authors agree with Mr. Feltner that the strain hardening exponent is better defined by considering the plastic range only. The variation in the slope n with stress value, as indicated by Mr. Feltner, is also reported in other investigations.

MR. ROBERT A. HELLER.[3]—The triaxial state of stress at the root of the fatigue crack may be responsible for the apparent high mechanical strength of specimens tested to 80 per cent of their fatigue life. Tension tests on notched specimens show that their ultimate strength may be increased by as much as 30 per cent above that of smooth specimens.

MR. HERBERT T. CORTEN.[4]—The measurement of ductility, the per cent reduction of area, constitutes an average value for one cross-section of the specimen. Do the authors interpret the differences measured in ductility as resulting from the presence of a number of localized fatigue damage areas or cracks?

A second point is concerned with the use of an average cycle ratio to estimate the damage in a specimen. Since the life of a particular specimen is unknown, the computation of the cycle ratio must be based on the average life of a large number of specimens. Thus, the true cycle ratio for a given specimen may be in error as much as 50 per cent or more. Consequently some of the irregularities of trends noted in connection with Table III may result from the fact that while the average cycle ratios were say 40 per cent and 80 per cent, the true cycle ratios for these specimens may be equal or in reverse order.

MR. MARIN.—The differences in ductility as measured by the reduction in area would apparently represent the influence of a number of localized fatigue damage areas. The improvement in results suggested by Mr. Corten is justified. However, this would require an appreciable increase in the number of specimens.

---

[3] Assistant Professor, Columbia University, New York, N. Y.

[4] Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

MR. OWEN RICHMOND.[5]—The authors concluded that prestressing usually decreased hardness while it increased the tensile strength. Since hardness and tensile strength are both measures of resistance to plastic flow it seems unusual that one of these properties should increase while the other decreased. Do the authors have an explanation for this?

MR. MARIN.—In answer to this question the changes are so small that it is difficult to make any conclusions about this observation.

---

[5] Research Engineer, E. C. Bain Laboratory for Fundamental Research, U. S. Steel Research Center, Monroeville, Pa.

REFERENCES

(1) C. E. Feltner and JoDean Morrow, "Micro-Plastic Strain Hysteresis Energy as a Criterion for Fatigue Fracture," *TAM Report No. 576*, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill., May 1959.

(2) T. H. H. Pian and R. D'Amato, "Low-Cycle Fatigue of Notched and Unnotched Specimens of 2024 Aluminum Alloy Under Axial Loading," *WADC Technical Note 58-27*, Feb. 1958.

(3) J. R. Low and F. Garofalo, "Precision Determination of Stress-Strain Curves in the Plastic Range," Soc. of Experimental Stress Analysis, Vol. IV, No. II (1947).

(4) C. E. Feltner, "The True Stress-Strain Tension Test—Its Value to Engineering Research," Unpublished Research Report, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill., March 1959.

SOME OBSERVATIONS ON THE PLASTIC WORK REQUIRED TO FRACTURE STAINLESS STEEL UNDER CYCLIC LOADING*

By D. E. Martin[1] and J. Brinn[1]

Synopsis

Controlled-load, low-cycle, axial fatigue tests were conducted on AISI type 347 stainless steel at 1000 F. Periodic records of the stress-strain behavior of the specimens were obtained by means of an oscilloscope camera. The results were analyzed in terms of the plastic strain per cycle and the total plastic work required to fracture the specimens. The data are compared with results of prior investigations of strain cycling, and a tentative criterion of fatigue fracture is discussed.

In recent years interest in low-cycle fatigue has been increasing, primarily because of advanced engineering projects involving members subjected to comparatively few but severe thermal loads.

A number of interesting papers **(1–6)**[2] have been published on this subject which have not only provided significant information pertaining to low-cycle fatigue design but have also contributed to a better understanding of the fatigue mechanism. Coffin **(4)** has demonstrated, under conditions of strain cycling, that plastic strain per cycle is a much more significant parameter than stress to associate with cyclic life. He described his results in the rather simple equation:

$$N^{\frac{1}{2}}\Delta\epsilon_p = C \text{............} (1)$$

where:

N is the number of cycles to fracture,

$\Delta\epsilon_p$ is the plastic strain per cycle, and

C is a material constant which is temperature dependent.

It is particularly interesting to note that the value of C determined in cyclic testing agreed rather well with the value that results from a static test, where $N = \frac{1}{4}$ and $\Delta\epsilon_p$ is measured from the true strain at fracture.

From studies of sequential loading, Coffin also demonstrated that the Miner hypothesis **(7)** of linear damage could not be applied to strain cycling. In sequential loading in which the applied total strain range was initially about 0.6 per cent for a number of cycles and was then increased to about 1 per cent for the remainder of cycles to fracture, he noted that the Miner sum[3]

$$\left(\Sigma \frac{n_i}{N_i}\right)$$

was greater than unity. Under reversed conditions in which the strain was initially 1 per cent and then reduced to 0.6 per cent he noted that the Miner sum was less than unity. This observation is typical for cumulative damage studies (8) and unfortunately demonstrates the most significant problem existing in the area of fatigue of metals: No adequate measure of fatigue damage exists. Until some measure or index of damage is obtained so that the fatigue mechanism can be more adequately described, design problems involving complex stress and temperature histories will be exceedingly difficult.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Research Engineers, General Motors Research Laboratories, Warren, Mich.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] Miner suggested that the sum of the cycle ratios should be equal to unity. $\Sigma \frac{n_i}{N_i}$, where n_i is the number of applied stress cycles at the ith stress level and N_i is the number of cycles required for fracture at that stress level.

In a discussion of Coffin's paper (4), Sinclair referred to earlier work on fatigue of brass (9) in which he noted that the total work under the true stress-strain diagram was independent of the initial state of the material. With this in mind and noting the consistent slope $(-\frac{1}{2})$ of Coffin's plastic strain - cyclic life diagrams, he suggested that the failure of the specimens might represent the attainment of a limiting amount of plastic work. If this were indeed the case, then a measure of the plastic work per cycle would provide an index of fatigue damage.

In the past, studies of hysteresis in fatigue have not always indicated a positive correlation with failure. For example hysteresis has been observed (10) in metals beneath the endurance limit. That is, a finite hysteresis loop occurs in members with presumably infinite life. It must be noted however that the occurrence of hysteresis below the nominal endurance limit does not necessarily mean that plastic work is not an index of damage. Hysteresis is not comprised of plastic work alone. Hysteresis measured in low-stress, long-life fatigue is primarily due to other energy dissipation forms such as diffusion and ferromagnetism. Further, the existence of an endurance limit may be associated with metallurgical changes (11) rather than a stress beneath which no damage occurs.

Recently, Gerberich (12) has qualitatively related plastic work rates to cumulative damage in fatigue. In sequential load testing he noted that if the stress (stress cycling) was first high and then low, the Miner sum was less than unity; and if the stress was first low and then high, the Miner sum was greater than unity. He also noted that in the first case (overstressing) the plastic work absorbed per cycle at the second stress level was greater (by virtue of the overstress) than it would have been for constant stressing at the second level. For the understressing, the work rate was less at the second level. Qualitatively this would explain the variations in the Miner sum. A similar argument was presented for strain cycling.

Although a number of investigations have been devoted to the study of hysteresis in fatigue, little has been accomplished in the realm of low-cycle fatigue under conditions of uniform strain distribution (direct stress or axial loading).

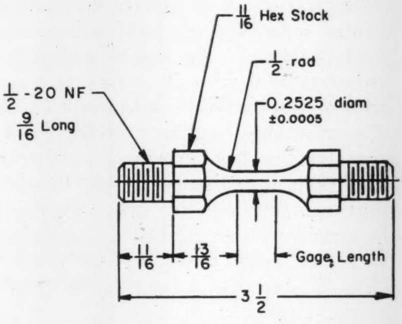
PURPOSE AND SCOPE

The purpose of this investigation was to examine plastic work as a possible index of low-cycle fatigue damage and to compare the plastic strain *versus* cyclic life behavior under conditions of load cycling with the same behavior in prior investigations conducted under controlled strain cycling.

Thirty specimens of AISI type 347 stainless steel were subjected to completely reversed, axial-load fatigue tests at 1000 F and 140 cpm. Periodically throughout 17 of these tests, simultaneous measurements of axial load and deformation were obtained. Measurements of plastic strain and plastic work from these records were used in the analysis of cyclic life.

MATERIAL AND SPECIMENS

The material employed in this investigation was AISI type 347 stainless steel. All specimens were machined from $\frac{11}{16}$-in.


11/16 Hex Stock
1/2 rod
1/2 -20 NF
9/16 Long
0.2525 diam
±0.0005
11/16
13/16
Gage Length
3 1/2

Note: All dimensions are in inches.

Fig. 1.—Fatigue Specimen.

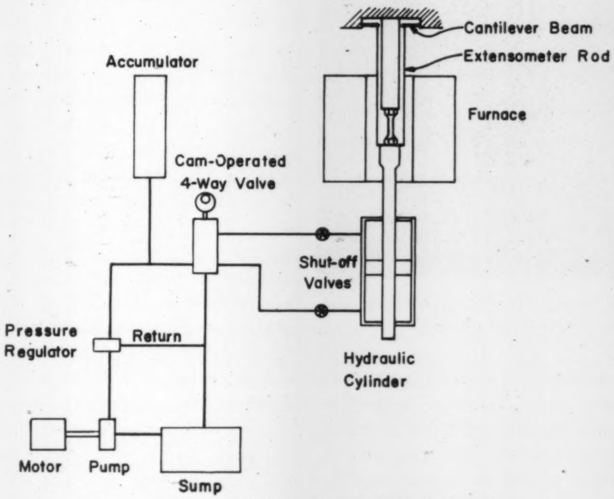

Cantilever Beam
Extensometer Rod
Accumulator
Furnace
Cam-Operated
4-Way Valve
Shut-off
Valves
Pressure
Regulator
Return
Hydraulic
Cylinder
Motor
Pump
Sump

Fig. 2.—Schematic Drawing of Testing Machine.

hexagonal stock, heated to 2000 F for 15 min, and water quenched. Final dimensions (Fig. 1) were obtained by grinding on centers after rough machining to within 0.006 in. To avoid buckling of the high-stress specimens, a short, $\frac{1}{2}$-in. gage length was employed, while for specimens subjected to lower stress, a 1-in. gage length was used to facilitate more accurate measurement of plastic strain and plastic work. The two size groups were subjected to overlapping stress magnitudes so that the influence of the gage length could be evaluated.

Test Machine and Instrumentation

The axial-load testing machine shown schematically in Fig. 2 was designed for controlled force-amplitude cyclic loading. The hydraulic pump supplied the desired pressure to the accumulator, and this pressure was alternately introduced into the cylinder on first one, and then the other side of the piston at 140 cpm by means of the cam-operated, four-way valve.

A three-zone, split electric furnace in conjunction with two thermocouples welded to the fillet radii of the specimen provided a constant temperature along the gage length of 1000 ± 15 F.

Although the applied load was indicated by the controlled hydraulic pressure, a wire-strain-gage load cell was used for load measurement.

Axial deformations were transmitted outside the furnace by extensometer rods and measured by means of cantilever-beam transducers. The axial displacements of the extensometer rods deformed the elastic cantilever beams which were provided with two bakelite strain gages for temperature compensation. Although false strain readings result from relatively small temperature variations in the extensometer rods during the hunting oscillations of the furnace control, these false strains are not significant for the time period ($\frac{1}{2}$ sec) corresponding to one load cycle.

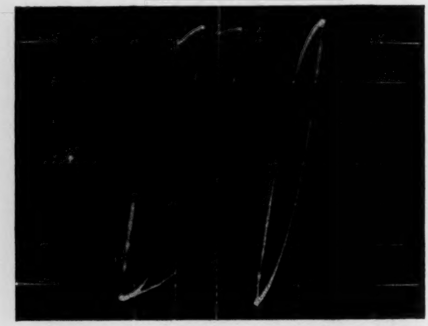


FIG. 3.—Hysteresis Loops. (*Left*) First and second cycles, (*Right*) 100th cycle.

The extensometer rods and cantilever beams, along with the load cell, provided the simultaneous load-deformation signals from which the dynamic stress-strain behavior of the specimens was recorded. These signals were amplified by d-c amplifiers and shown simultaneously on the horizontal and vertical axes of an oscilloscope. The hysteresis loops, Fig. 3, were obtained with a Polaroid oscilloscope camera. The accuracy of individual measurements of some of the plastic strains from these diagrams was approximately ±10 per cent. However, from 10 to 25 measurements were made for each specimen, so the average values were more accurately determined. The hysteresis areas were measured by means of a planimeter and here also for some of the smaller areas the per cent errors were large; however large numbers of measurements minimized the errors. It is not known what part of these hysteresis areas is attributable to plastic strains; however for the low-cycle and high-stress conditions investigated it was assumed that contributions by other forms of hysteresis were negligible in comparison with the large variations with stress (Fig. 7) observed in the total hysteresis areas. Examination of the hysteresis loops revealed rather sharp ends. Had significant anelastic strains occurred, they would have been evident in the form of blunted peaks on the hysteresis loops, as the load-time history was such that the maximum and minimum loads were sustained for about $\frac{1}{10}$ sec. The sustained load was revealed in the photographs of the hysteresis loops in the form of a dot, due to the prolonged exposure on the film in the extreme load positions.

TEST RESULTS

Fatigue tests were conducted on 30 specimens, and on 17 of these, stress-strain behavior was recorded. The results of these 30 tests are summarized in the conventional *S-N* diagram shown in Fig. 4. Here, there appears to be no significant difference between the $\frac{1}{2}$-in. and 1-in. gage-length specimens. If, however, we examine Fig. 5, which is the plastic strain diagram of the type employed by Coffin (4), it appears that the $\frac{1}{2}$-in. specimens were somewhat stronger on the basis of strain. It is interesting to note that the $\frac{1}{2}$-in. specimens (the gage length employed by Coffin) are in excellent agreement with Coffin's results (interpolate to 1000 F) which were obtained on specimens subjected to controlled total strain range rather than controlled load. The plastic strains per cycle plotted in this diagram were the average values taken from the oscilloscope diagrams.

The width of the hysteresis loop for each specimen was plotted against the corresponding number of cycles, and an average value obtained from the resulting graph.

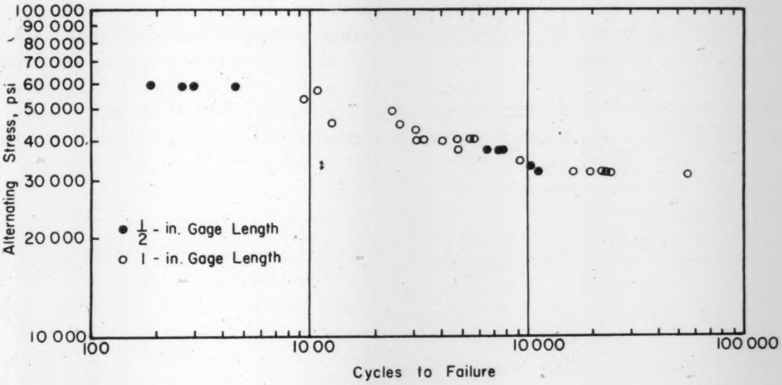


FIG. 4.—*S-N* Diagram.

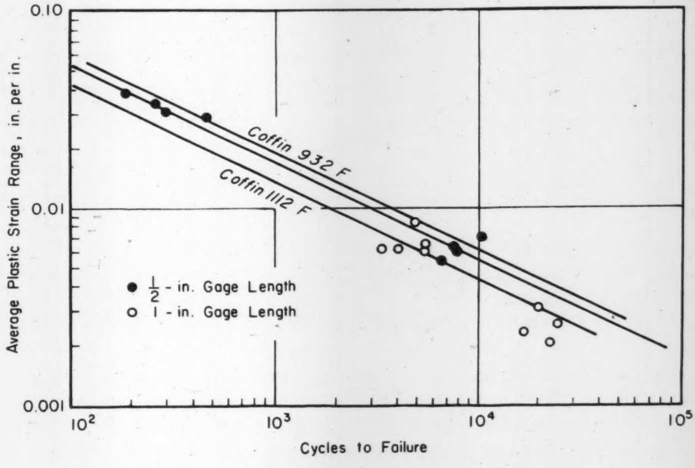


FIG. 5.—Plastic Strain *versus* Cycles to Failure.

The appearance of the strain per cycle *versus* cycles diagrams was very similar to the corresponding curves of plastic work per cycle illustrated in Fig. 6. Here the plastic work per cycle (hysteresis area) has been plotted *versus* the number of cycles. On all specimens at all stress levels the plastic work on the first few stress cycles was large and decreased rapidly before increasing again on subsequent cycles. This initial dip evident in Fig. 6 was followed by either a constant or gradually increasing trend in the cases

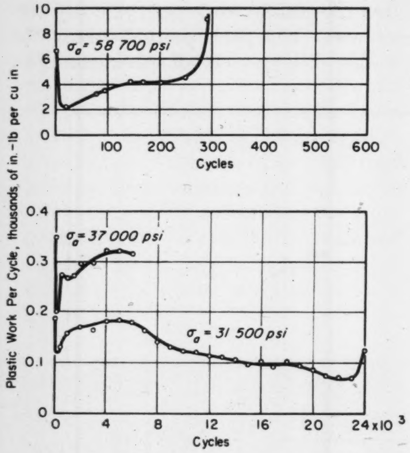


FIG. 6.—Plastic Work per Cycle *versus* Number of Cycles.

of high stress and a general decrease in hysteresis in the lower stress specimens until the onset of fracture, at which time the areas grew very rapidly. The area under this diagram is the total plastic work required to produce fracture and is plotted against the applied stress for each of 17 specimens in Fig. 7. Although there is considerable scatter in these data, there can be no question that the average total plastic work required to fracture these specimens is not constant but rather decreases with increasing stress. The decreasing trend may at first imply that plastic work cannot be regarded as an index of fatigue damage; however further consideration should be given to factors evaluated in the following discussion.

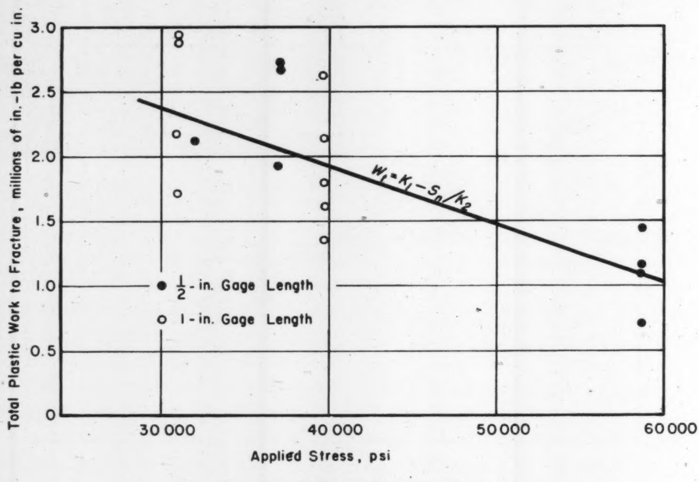


FIG. 7.—Total Plastic Work to Fracture.

DISCUSSION

Most investigations concerning cumulative damage in fatigue have assumed that fracture occurs when a critical amount of damage has been achieved and that this amount is a constant. Miner (7) for example implies this in his linear damage hypothesis described by the cycle ratio, n_i/N_i. When we examine fracture surfaces of fatigue specimens, however, we realize that this is not the case. In a prior investigation (13) concerning crack propagation, it was shown that the critical crack length at fracture was dependent upon the applied load and that the crack length one cycle prior to fracture was such that the nominal stress on the uncracked cross-section of the specimen was constant for all loads on the fracture cycle. This can be expressed as follows:

$$S_f = \frac{P_f}{A_f} = K \ldots\ldots\ldots\ldots (2)$$

where:

P_f is the load at fracture,

A_f is the fracture area or the uncracked cross-sectional area on the fracture cycle, and

S_f is the nominal fracture stress, a constant.

The fracture area A_f can be expressed in terms of the original specimen area A_0 and the reduction in area A_{1f} formed by the fatigue crack of length $1f$:

$$A_f = A_0 - A_{1f} \ldots\ldots\ldots\ldots (3)$$

or:

$$S_f = K = \frac{P_f}{A_f} = \frac{P_f}{(A_0 - A_{1f})} \ldots\ldots (4)$$

or:

$$A_{1f} = A_0 - \frac{P_f}{K} \ldots\ldots\ldots\ldots (5)$$

This same idea has been noted qualitatively for years in examinations of fracture surfaces such as those illustrated in a paper by Demer (**14**, Figs. 39 and 41). The results of Nordmark and Eaton (**15**) indicate that the occurrence of a fatigue crack in several aluminum alloys reduced the static strength by an amount greater than would be indicated by the reduction in cross-sectional area. These observations, which are contrary to Eq 2, can be explained in terms of the Griffith theory of fracture, or by means of the crack driving force described by Irwin (**16**). Unfortunately the final fracture of a member in which a crack exists will be complicated by the stress concentrating factor of the crack and the features of stability discussed by Irwin. It can be concluded, however, that the amount of fatigue damage required to produce fracture under a given load is not constant but rather is dependent among other things upon the fracture load. This is true, no matter what the fatigue damage may be. If we use crack length as an index of damage, then for ductile metals Eq 5 is applicable. Crack length, however, cannot be regarded as a good index of fatigue damage, partially because of our inability to detect and measure cracks accurately early in the fatigue process.

If we examine plastic work as a possible index of fatigue damage, then it is clear that we must describe the total plastic work required to produce fracture at least in terms of the fracture load. As a first approximation, Eq 5 was employed. The crack length or cracked area A_{1f} was replaced by the total plastic work (as measured from the curves in Fig. 6), giving:

$$W_t = K_1 - \frac{S_n}{K_2} \ldots\ldots\ldots\ldots (6)$$

where:

S_n is the nominal stress at the time of fracture, based on the original specimen dimensions and the fracture load, and

K_1 and K_2 are experimentally determined constants.

The favorable agreement of this equation with the data in Fig. 7 is not sufficient to ensure the validity of using plastic work as an index of damage, since the analogy between crack length and plastic work is only qualitative; however it is certain that the damage required must decrease with the load at fracture. The use of an *S-N* diagram to predict failure requires that the loading history follow one constant stress path. The use of Fig. 7 permits one to say that if one reaches the line defined by Eq 6 failure will occur. No unique path to this line is prescribed.

Accordingly, Eq 6 is regarded as a starting point for further investigations. It is proposed to study further the nature of plastic deformation and plastic work on simple, single-phase materials under room temperature conditions, on large numbers of specimens so that a statistical evaluation of sequential loading can be accomplished.

SUMMARY AND CONCLUSIONS

Axial-load, low-cycle fatigue tests were conducted on AISI type 347 stainless steel at 1000 F under conditions of controlled load. Throughout these tests, axial load and strain signals were recorded in the form of stress-strain hysteresis diagrams. Examination of these hysteresis loops showed that although the plastic strain underwent large changes throughout the life of most specimens, the average plastic strain per cycle for a given cyclic life was in good agreement with the results of Coffin (**4**) for controlled strain cycling.

The total plastic work required to bring the specimens to the point of fracture decreased with increasing load. Examination of the data and consideration of prior investigations suggested the tentative criterion of low-cycle fatigue fracture expressed in the following equation:

$$W_t = K_1 - \frac{S_n}{K_2}$$

where K_1 and K_2 are constants and W_t is the total plastic work required to produce fracture under an applied nominal stress (or load) S_n. Although the true conditions governing fracture even under the restricted conditions of low-cycle fatigue may be more complicated that implied by this equation, it is regarded as a starting point for further studies of the mechanism of fatigue. It is anticipated that even if the plastic work per cycle is an index of fatigue damage, additional factors such as crack propagation may have to be incorporated in a final description of the failure process.

Acknowledgment:

This investigation was conducted at the Research Laboratories of the General Motors Corp. as part of a program intended to study the failure mechanism of metals. The authors wish to acknowledge the help of Mr. C. K. Kroell in the initial design and development of the test equipment. They wish also to express their gratitude to Mr. R. Siegrist for the energetic manner in which he conducted the tests, and for the excellent judgment he displayed in his photographic work.

REFERENCES

(**1**) Harry Majors, Jr. "Thermal Shock and Fatigue, A Literature Survey," *Bulletin No. 4*, Bureau of Engineering Research, College of Engineering, Univ. of Alabama.

(**2**) H. R. Hardrath and W. Illg, "Fatigue Tests of Stresses Producing Failure in 2 to 10,000 Cycles," *Technical Note 3132*, Nat. Advisory Comm. Aeronautics, Jan., 1954.

(**3**) A. C. Low, "Short Endurance Fatigue," International Conference on Fatigue of Metals, Session 2, Paper 15, Nov.; 1956.

(**4**) E. E. Baldwin, G. J. Sokol, and L. F. Coffin, "Cyclic Strain Fatigue Studies on AISI Type 347 Stainless Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, pp. 567–586 (1957).

(**5**) S. I. Lui, J. J. Lynch, E. J. Ripling, and G. Sachs, "Low Cycle Fatigue of Aluminum Alloy 24S-T in Direct Stress," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 175, p. 469 (1948).

(**6**) D. A. Douglas, R. W. Swindeman, "The

Failure of Structural Metals Subjected to Strain Cycling Conditions," Paper 58-A-198, Am. Soc. Mechanical Engrs. 1958 Annual Meeting.

(7) M. A. Miner, "Cumulative Damage in Fatigue," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 67, pp. 159–164 (1945).

(8) H. T. Corten and T. J. Dolan "Cumulative Fatigue Damage," International Conference on Fatigue of Metals, Am. Soc. Mechanical Engrs., New York, 1956.

(9) G. M. Sinclair and W. J. Craig, "Influence of Grain Size and Work Hardening and Fatigue Characteristics of Alpha Brass," *Transactions*, Am. Soc. Metals, Vol. 44, pp. 929–948 (1952).

(10) B. P. Haigh, "Hysteresis in Relation to Cohesion and Fatigue," *Transactions*, Faraday Soc., Vol. 24, pp. 125 (1928).

(11) F. C. Rally and G. M. Sinclair, "Influence of Strain Aging on the Shape of the *S-N* Diagrams," Theoretical and Applied Mechanics, *Report No. 87*, Univ. of Illinois, Urbana, Ill. (1959).

(12) W. W. Gerberich, "The Phenomena of Cumulative Damage in Stress Cycling and Strain Cycling Fatigue," Syracuse University Research Inst. Report No. Met 575-594T3, April 1959.

(13) D. E. Martin and G. M. Sinclair, "Crack Propagation Under Repeated Loading," Third Nat. Congress of Applied Mechanics, Brown Univ. (1958).

(14) L. J. Demer, "Interrelation of Fatigue Cracking, Damping, and Notch Sensitivity," *Technical Report 56-408*, Wright Air Development Center, March, 1957.

(15) G. E. Nordmark, and I. D. Eaton, "Effect of Fatigue Crack on Static Strength," Nat. Advisory Comm. Aeronautics, *Technical Note 1428*, May, 1957.

(16) G. R. Irwin, "Fracture Strength," *Progress Report*, National Research Laboratory, Nov., 1957.

DISCUSSION

MR. BENJAMIN J. LAZAN.[1]—Similar studies on total energy to fracture were undertaken some years ago except at low stress or strain amplitudes—amplitudes in the conventional fatigue range. This work at low strain amplitudes led to a conclusion similar to that found by the authors at high strain amplitudes: namely, the lower the strain amplitude (or stress amplitude) the higher is the total energy to fracture.

An effort was made in the work at low strain amplitudes to determine if under mixed loadings the energy to the fracture might be used as a criterion for predicting the point of fatigue failure. This did not prove to be a suitable criterion at the low strain amplitude since many different total energies to fracture were observed. The suitability of such a criterion at high strain amplitude still remains to be determined.

The authors imply, I believe, that the presence of hysteresis indicates that damage is taking place. It should be pointed out that significant hysteresis may be present at stress levels well below the fatigue limit. There are a large variety of hysteretic damping mechanisms, many of which are not apparently damaging in the fatigue sense.

MR. D. E. MARTIN (*author*).—I agree wholeheartedly that at stress levels beneath the endurance limit, energy is being absorbed in a fashion that may not be damaging. We have hypothesized that the plastic work is associated with damage and that the hysteresis due to anelastic strains is not. Because the plastic strains are predominant for high stress conditions, we feel it may be possible to relate hysteresis measurements to low-cycle fatigue damage, although, as Mr. Lazan has pointed out, this has not been possible for lower stress levels where anelastic strains become significant. Although it is quite obvious that all of the hysteresis that occurs at stress levels beneath the fatigue limit is not damaging, I do not believe that one can definitely say that the fatigue limit is a stress beneath which no damage occurs. There has been a great deal of evidence to show that the endurance (or fatigue) limit is due to a metallurgical change. It seems entirely possible that damage is still going on at stresses beneath the endurance level, but not at a rate that produces failure in a measurable period of time.

---

[1] Professor of Materials Engineering, University of Minnesota, Minneapolis, Minn.

MESSRS. JODEAN MORROW AND C. E. FELTNER[2] *(by letter)*.—A similar set of tests at room temperature using SAE 4340 steel (Rockwell hardness C 30) were completed at Illinois this spring (1).[3] An analysis based on plastic strain hysteresis as a criterion for fatigue fracture was used. The predicted and experimental *S-N* curve agreed fairly well, as shown in the accompanying Fig. 8, giving hope that an energy approach to fatigue holds some promise.

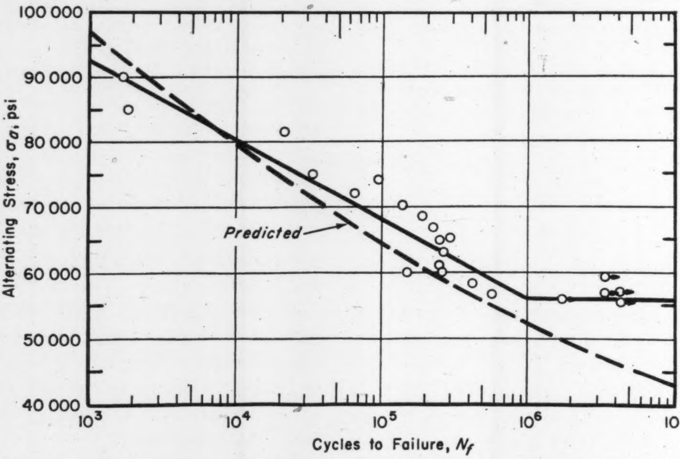


FIG. 8.—Comparison of Test Results with Analysis (Reference (1)).

The data presented in this paper offer a welcome opportunity to apply such a criterion to a different material at elevated temperature in the low-cycle fatigue region.

Hysteresis energy has been measured for cyclic stresses as low as 10 psi (2). As the stress amplitude is increased, the hysteresis energy per cycle increases as a power function of the stress (3) or

$$\Delta W = \text{hysteresis energy per cycle} = K\sigma^m \quad (1)$$

Equation 1 holds for stresses well below the fatigue limit with measured values of *m* ranging between 2 and 3 (4). Near the fatigue limit, the hysteresis energy per cycle increases more rapidly with increasing stress (*m* increases). For high stresses, *m* approaches a constant again, the value ranging between 3 and 20, depending on the material and test temperature.

When the logarithm of both sides is taken, Eq 1 is transformed into a linear function of the logarithm of the two variables,—stress and hysteresis energy per cycle. The exponent *m* is the slope of a log-log plot of the two variables. Such a plot will have two linear regions, with a small slope at low stresses and a large slope at high stresses, with a transition zone between. Such a dependence of hysteresis energy on stress is borne out by experimental observations such as those reported by Mason (5) and shown in Fig. 9.

---

[2] Associate Professor and Graduate Fellow, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[3] The boldface numbers in parentheses refer to the list of references appended to this discussion.

The authors imply that hysteresis is only partly damaging in a fatigue sense. The writers believe that the damaging portion should be associated with the linear region of Fig. 9 at high stresses (plastic-fatigue region), and the nondamaging portion should be associated with the linear region of Fig. 9 at low stresses (anelastic region). At very low stresses a large amount of total hysteresis energy can be accumulated without fatigue fracture since in this region the nondamaging type of hysteresis dominates. At high stresses the damaging plastic strain hysteresis dominates permitting less total hysteresis to be accumulated before fatigue fracture occurs. A plot of total work to fracture such as the authors' Fig. 7 should then have a vertical asymptote at the fatigue limit and a horizontal asymptote at some minimum value of the total energy (for example, the area under a static true stress-strain curve). Thus, the straight line shown by the authors in Fig. 7 is only valid inside this range. Furthermore, the scatter of the total energy data in Fig. 7 is as large as the range of the solid line.

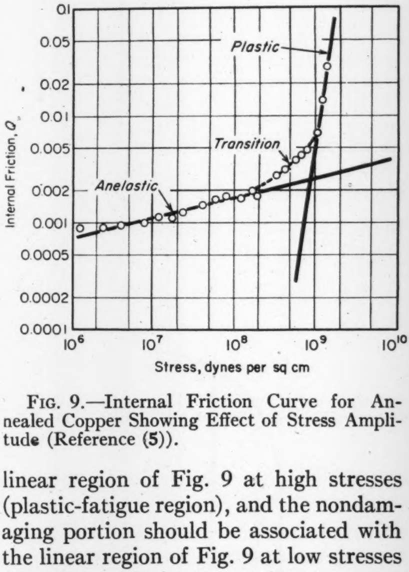


FIG. 9.—Internal Friction Curve for Annealed Copper Showing Effect of Stress Amplitude (Reference (5)).

A criterion for fatigue fracture based on hysteresis energy should consider of prime importance the damaging plastic strain hysteresis energy which can be most readily identified at high stresses. Other forms of nondamaging hysteresis energy (anelastic effects) which dominate at low stresses, only tend to obscure the plastic strain hysteresis and confuse the problem.

The aim of the analysis presented in reference (1) was to explore a method of predicting fatigue behavior from only a knowledge of the magnitude of plastic strain hysteresis energy per cycle. This was done by integrating for the area within the static true stress - plastic strain loop, obtaining an equation for the plastic strain hysteresis energy per cycle as a function of the stress amplitude. This amount of plastic strain hysteresis energy per cycle was considered not to change during cyclic loading. Fracture was assumed to occur when an amount of plastic strain hysteresis energy was accumulated equal to the energy to cause fracture in a static tension test.

In making such simplifying assumptions several experimentally established facts concerning hysteresis are ignored, such as the effect of stress history. By so doing it is possible to keep the analysis simple enough that an end result (predicted *S-N* curve) can be obtained which is free of experimentally determined constants, except those determined in the static tension test. In addition, errors introduced into the analysis because of the simplifications show up as errors in predicted life. Thus, if the cyclic hysteresis energy per cycle is only half that

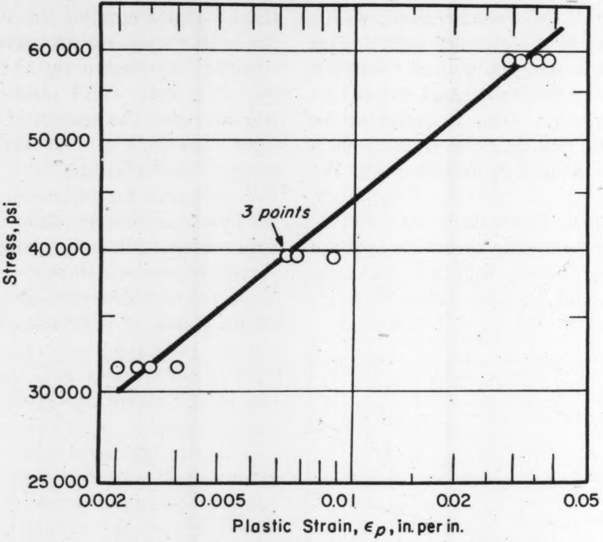


FIG. 10.—Logarithmic Stress - Plastic Strain Curve for 347 Stainless Steel.

assumed in the analysis, a factor of two error will appear in the predicted life. For this reason, small errors are not too significant, since it is not uncommon to measure scatter in fatigue lives of a factor of five for identical specimens at the same stress level.

Further details of the analysis of reference (1) will not be given here, but the same approach will be applied to the data for stainless steel at elevated temperature.

First a relationship must be established relating the plastic strain hysteresis energy per cycle to the stress amplitude. This was done by plotting corresponding values of alternating stress and plastic strain range from the reported data. Using the log-log plot shown in Fig. 10 permits an equation of the following form to be written for plastic strain, ϵ_p, as a function of σ, the stress amplitude:

$$\epsilon_p = \epsilon_c\left(\frac{\sigma}{\sigma_c}\right)^{1/n} \qquad (2)$$

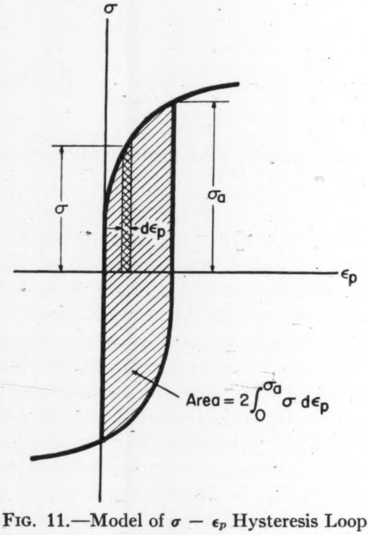


FIG. 11.—Model of $\sigma - \epsilon_p$ Hysteresis Loop.

where ϵ_c and σ_c are convenient corresponding values of plastic strain and stress and n is the slope of the log-log plot shown in Fig. 10.

The data of Fig. 10 are replotted schematically in Fig. 11 to show how the plastic hysteresis energy per cycle for some stress amplitude, σ_a, can be obtained by integration as follows:

hysteresis energy per cycle

$$= \frac{2\epsilon_c}{n(\sigma_c)^{1/n}} \int_0^{\sigma_a} \sigma^{1/n} d\sigma \quad . . (3)$$

For the high stresses used in this investigation, the hysteresis energy is predominantly damaging plastic strain hysteresis. For this reason all the hysteresis energy per cycle given by Eq 3 will be considered to contribute to fatigue failure. Also, for simplicity, Eq 3 will be assumed to hold throughout the test (that is, the hysteresis is not dependent on stress history). Judging from the authors' Fig. 6 an error of perhaps a factor of 2 might be introduced by this assumption. It is also assumed that fatigue failure at N cycles represents the attainment of some limiting amount of total plastic strain hysteresis energy, W_p, and that this total energy quantity is constant.

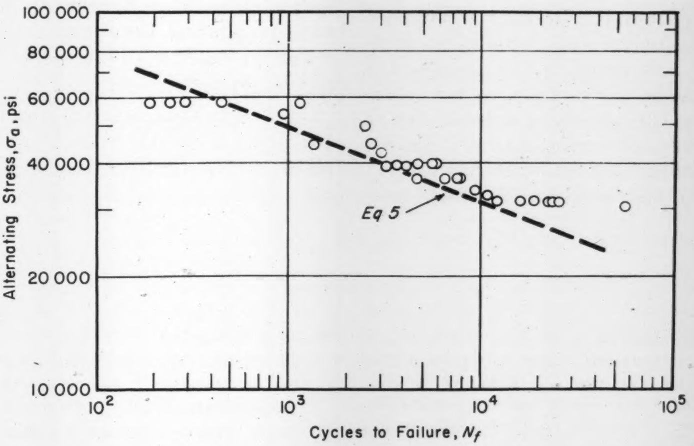


FIG. 12.—Analysis of Reference (1) Applied to Data of Martin and Brinn.

In equation form,

$$W_p = N\left(\frac{\text{hysteresis energy}}{\text{cycle}}\right)$$

$$= \frac{N 2\epsilon_c}{n(\sigma_c)^{1/n}} \int_0^{\sigma_a} \sigma^{1/n} d\sigma \quad . . (4)$$

Transposing terms, integrating, and substituting in limits,

$$\sigma_a^{1/n + 1} N = W_p \frac{(1 + n)\sigma_c^{1/n}}{2\epsilon_c} \quad . . . . . (5)$$

The fatigue data reported in Fig. 4 is replotted in Fig. 12 with Eq 5 shown as a dashed line. Values of $n = 0.25$, $\epsilon_c = 0.0063$, and $\sigma_c = 40{,}000$ psi, were obtained from Fig. 10. A value of $W_p = 1{,}000{,}000$ lb-in. per cu in. was chosen as representative of the total plastic work to fracture at the highest stress investigated. The agreement between Eq 5 and the cyclic data is very good.

It is not our intention to imply that Eq 5 can be used *in general* to predict fatigue lives. However, we do feel that a refinement of such an analysis will be possible as more fatigue data becomes available such as reported in this paper wherein strains as well as stresses are measured during cyclic loading. The analysis implies that stress is no *real* criterion for fatigue failure but rather is the significant variable incidental to a plastic hysteresis energy criterion.

MESSRS. D. E. MARTIN AND JULE BRINN (*authors' closure*).—The authors are grateful to Messrs. Morrow and Feltner for their interesting discussion and further analysis of our data.

The method of predicting fatigue life suggested by Morrow and Feltner is particularly interesting in that it would seem to require only a static tension test; however as was noted in the discussion it would seem appropriate to refine the analysis. The agreement noted in Fig. 12 would be considerably better if instead of regarding the plastic work a constant, Eq 6 had been employed. It would seem that it is possible to consider the total plastic work to fracture as a constant for the purpose of approximately predicting fatigue life; however, there can be no doubt that it is not. Although, as was pointed out in the discussion, the scatter of the total energy in Fig. 7 is indeed as large as the range of the solid line, this should not be surprising for fatigue data, where lives at identical stresses vary by orders of magnitude.

The authors agree that a curve of total work would not be linear in the region of the fatigue limit; however, it does not seem possible to draw any conclusions about the "horizontal asymptote" which was suggested to exist at the higher stresses. The linear relation suggested in Fig. 7 was intended to describe the total plastic work in the low cycle fatigue range only.

REFERENCES

(1) C. E. Feltner and J. Morrow, "Micro-Plastic Strain Hysteresis Energy as a Criterion for Fatigue Fracture," *TAM Report No. 576*, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill., May 1959.

(2) C. Zener, "Elasticity and Anelasticity of Metals," University of Chicago Press (1948).

(3) B. J. Lazan, "Fatigue Failure Under Resonanti Vbration Conditions;" Fatigue, Chapter 2, Am. Soc. for Metals, Cleveland (1954).

(4) N. L. Person and B. J. Lazan, "The Effect of Static Mean Stress on the Damping Properties of Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 1399 (1956).

(5) W. P. Mason, "Internal Friction, Plastic Strain, and Fatigue in Metals and Semiconductors," Symposium on Basic Mechanisms of Fatigue, Am. Soc. Testing Mats., p. 36 (1958). (Issued as separate publication *ASTM STP No. 237*.)

TECHNIQUES AND EQUIPMENT FOR FATIGUE TESTING AT VERY HIGH FREQUENCIES*

By E. A. Neppiras[1]

Synopsis

A description is given of resonant ultrasonic vibrators used recently in high-frequency fatigue testing. Vibrators consist of three resonant sections: transducer, velocity transformer, and test specimen. Conventional types of electromechanical transducer—piezomagnetic, piezoelectric, and electrodynamic—can be used, operated in extensional, torsional, or flexural modes. A study of the design of resonant velocity transformers and test specimens for this work is reported. Practical systems have been operated at frequencies just above the audible limit—18,000 to 22,000 cps.

Apart from obvious advantages of speed and silent operation, ultrasonic testers are simple and economical to run, with a wide range of applications. These include dynamic-strength testing of metals, glass, plastics, adhesive bonds, and film adhesion; investigating non-linear behavior of metals, rubbers, and plastics; and wear tests. The same basic vibrator and generator can be used for all such purposes.

Fatigue limits of metals at high frequency are rarely identical with those measured at low frequency; discrepancies up to 40 per cent have been observed for light alloys, steels and brasses. At high frequency, precautions must be taken to avoid overheating; suitable techniques are described.

Conventional fatigue testers are not designed to operate at frequencies above about 200 cps; the more usual speed is much lower—generally 25 or 50 cps. If only one machine is used, approximately a whole year is required to accumulate sufficient data to give a stress-endurance characteristic extending to, say, 10^8 reversals. A real need exists for an accelerated tester capable of giving results in hours rather than months or years. Apart from speeding routine tests, components in modern engineering are often subjected to high-speed stressing in service and a suitable testing technique for frequencies in the high sonic and low ultrasonic range is required. A further urgent reason for developing high-speed testers is to provide research tools for investigating the effect of speed of test on endurance. In this way, a fuller understanding of the basic mechanism of fatigue may be obtained.

Many investigators have worked at frequencies up to about 200 cps, since this range can be covered using commercially available testers. Jenkin[2] (**1**) and Jenkin and Lehmann (**2**) seem to have been the first to use high sonic frequencies. They used a resonant pneumatic vibrator to drive a small beam specimen into flexural resonance at frequencies up to 18,000 cps. Their apparatus could not easily be adapted to industrial testing and the results obtained by them rest on uncertain assumptions, making the technique open to criticism. More recently, research has been done at frequencies between 1000 and 5000 cps, using either pneumatic, motor, or electromagnetic drive to resonate specimens in flexure, torsion, or axial push pull (see for example: Hanstock and Murray (3); Thompson et al (4); Lomas et al (5); Wade and Grootenhuis (6)). Although the vibrators used by these workers are theoretically capable of modification to even higher frequencies, the practical difficulties are great. These equipments are also not easily adapted to industrial testing and are generally capable of driving specimens in only one mode of vibration.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Mullard Research Laboratories, Salfords, Surrey (England).

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

To cope with higher frequencies, it is necessary to revert to the types of vibrator commonly used in high-energy ultrasonics work. These are capable of producing large alternating stresses at frequencies from a few thousand to several million cycles per second when used with suitable focussing devices. The extension of fatigue testing to these very high frequencies has taken place during the past few years. In this paper, the design and performance of efficient vibrators for high frequency work will be discussed.

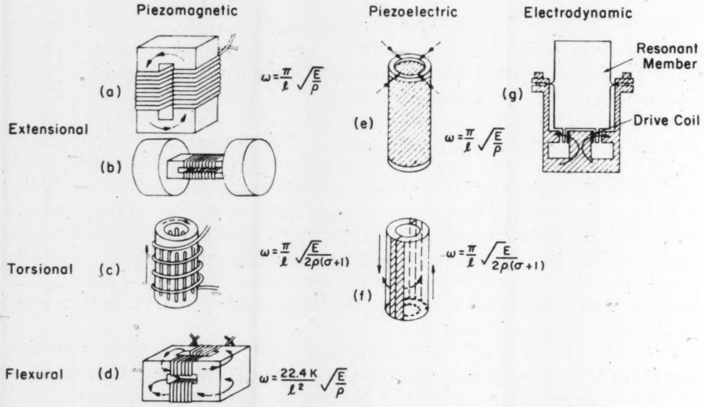

Piezomagnetic
Piezoelectric
Electrodynamic
Extensional
(a)
$\omega = \frac{\pi}{l}\sqrt{\frac{E}{\rho}}$
(b)
(e)
$\omega = \frac{\pi}{l}\sqrt{\frac{E}{\rho}}$
(g)
Resonant Member
Drive Coil
Torsional
(c)
$\omega = \frac{\pi}{l}\sqrt{\frac{E}{2\rho(\sigma+1)}}$
(f)
$\omega = \frac{\pi}{l}\sqrt{\frac{E}{2\rho(\sigma+1)}}$
Flexural
(d)
$\omega = \frac{22.4K}{l^2}\sqrt{\frac{E}{\rho}}$

FIG. 1.—Basic Transducer Types Used in Ultrasonic Testing.

E = Young's modulus, ρ = density, σ = Poisson's ratio, l = length or equivalent length, ω = angular frequency, K = radius of gyration of section about center line perpendicular to plane of bending. → polarization, ⇢ drive.

THE DRIVE VIBRATOR

There are three basic types of electromechanical ultrasonic transducers: the piezoelectric crystal, the piezomagnetic oscillator and the electrodynamic transducer. The strong need for a high-speed tester is emphasized by the fact that all three types have recently been used in experimental work in this field. Mason (7) first used crystal transducers in fatigue testing. He coupled vibrations to the specimen through a tapered metal stub serving as a velocity transformer; the specimen could also be tapered to give some stress amplification. The author has developed this technique using resonant magnetostriction and electrodynamic vibrators (8).

The need for mechanical focussing outside the transducer is dictated by the fact that the transducer material itself will generally not be capable of withstanding alternating stresses of the required magnitude. In some cases all the amplification required can be produced in the specimen itself, which is designed as a stress transformer. But generally it is more convenient to include at least one tuned velocity transformer between the transducer and specimen. The vibrator therefore consists essentially of three resonant sections: a transducer, one or more velocity transformers, and a specimen designed as a stress transformer. Ultrasonic testing also requires a variable-frequency oscillator-amplifier for transducer drive and some means of measuring the applied strain and counting strain reversals.

Vibrations may be required in any or all of the three useful modes. But testing in axial push-pull is accepted to be the most valuable since it is the only method that allows a large volume of material to be tested with uniform stressing over a specified test region. This discussion will therefore concentrate on this type, although methods of driving specimens in the other modes will also be indicated.

There are a number of transducer designs suitable for fatigue work. Basic information on types for extensional- and torsional-mode operation is available (9,10). Figure 1 shows diagrammatically those actually used in practical testers. The window-type piezomagnetic transducer of Fig. 1(*a*) is of ceramic ferrite or metal laminations (nickel, nickel-iron, or aluminum-iron) bonded into a rigid stack using an insulating cement. This is wound toroidally with a coil which carries direct-current polarization and alternating-current drive at the frequency of longitudinal resonance of the stack. The torsional transducer of Fig. 1(*c*) is either a bonded scroll of thin sheet metal or a ferrite tube, wound with two coils, as shown, providing flux in two perpendicular directions. The coils carry, respectively, direct and alternating current for polarization and drive. Resonant flexural vibrations are best obtained from two mechanically-coupled extensional-mode vibrators driven in opposite phases. The window-type construction can be used, the two windings being separated and either polarized or driven in opposite directions (Fig. 1(*d*)).

The piezoelectric ceramic transducers shown in Figs. 1(*e*) and (*f*) are plated tubes of barium titanate or lead zirconate titanate. For vibration in extensional and torsional modes they are polarized and driven electrically as shown. Frequency equations are given in Fig. 1.

The electrodynamic ultrasonic transducer is, in principle, similar to the ordinary moving-coil loudspeaker, with the cone replaced by a resonant metallic member. An efficient vibrator suitable for fatigue testing can be constructed to the St. Clair design (11). The cylindrical vibrator carries a flange on one face which rests in the gap of a pot magnet (Fig. 1(*g*)). The drive coil is wound on the central pole of the magnet and the alternating current induced in the flange (which constitutes a single-turn secondary in the radial field of the magnet) provides the longitudinal exciting force on the vibrator. The vibrator is supported on a diaphragm accurately located in the nodal plane.

In fatigue testing, piezomagnetic transducers show certain practical advantages over the other vibrators discussed above. Metallic piezomagnetic materials are robust and not easily damaged by rough handling. The problem of coupling efficiently to metal transformer stubs is also simplified—they can be soldered, brazed, or bolted. Certain recently developed, low-porosity piezomagnetic ceramics, although less robust, should also be valuable.

The useful frequency range is decided by the material and transducer type rather than the mode of vibration. Metallic piezomagnetic transducers are valuable in the range of 15 to 50 kcps, piezomagnetic ceramics between 15 and 200 kcps, and piezoelectric ceramics between 15 and 2000 kcps. But the very high frequencies, above 200 kcps, are not likely to be valuable in fatigue work because of the large elastic dissipation encountered and the small size of specimens. If piezomagnetic or piezoelectric transducers are required for frequencies in the audible range, below about 15 kcps, the best method is to use mass-loaded transducers, of the type shown in Fig. 1(*b*). The resonant frequency, ω, of a transducer with equivalent mass M and compliance C is given by $\omega^2MC = 1$. Addition of masses M_1 at the two end faces reduces the resonant frequency to ω_1, where $\omega_1^2(M + 2M_1)C = 1$.

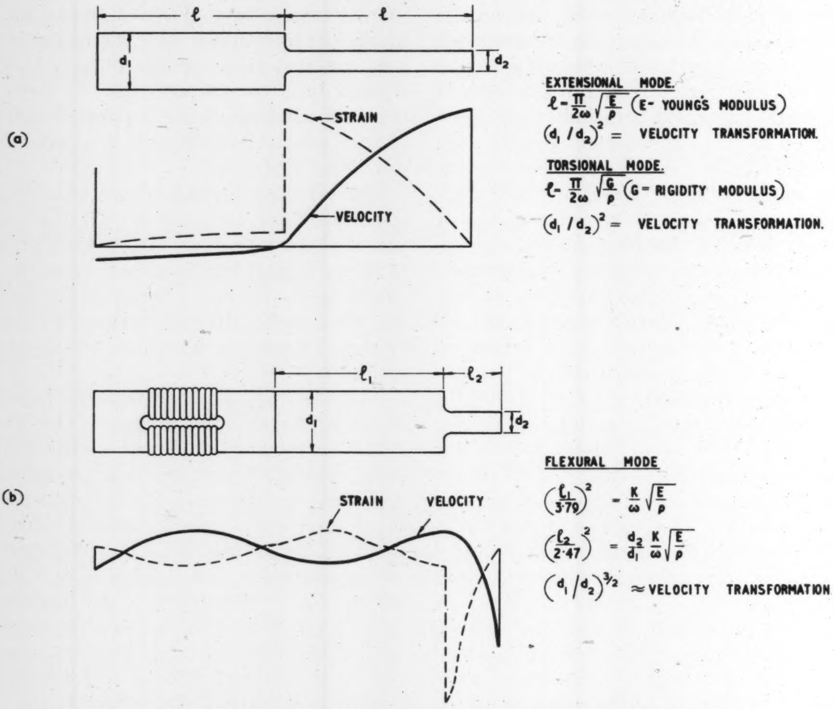
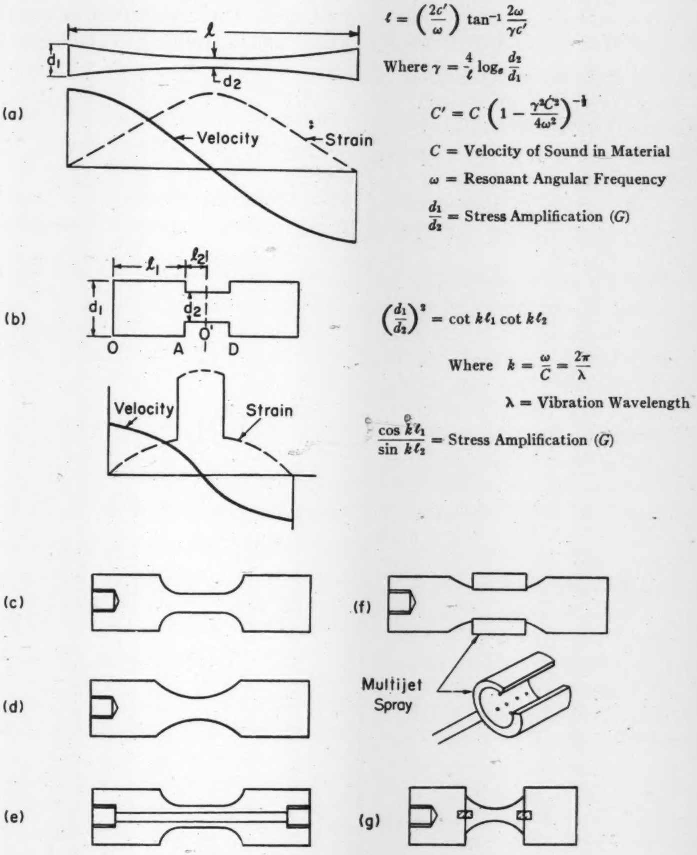

ℓ
ℓ
d_1
d_2
EXTENSIONAL MODE.
$\ell = \frac{\pi}{2\omega}\sqrt{\frac{E}{\rho}}$ (E = YOUNG'S MODULUS)
$(d_1/d_2)^2$ = VELOCITY TRANSFORMATION.
TORSIONAL MODE.
$\ell = \frac{\pi}{2\omega}\sqrt{\frac{G}{\rho}}$ (G = RIGIDITY MODULUS)
$(d_1/d_2)^2$ = VELOCITY TRANSFORMATION.
(a)
STRAIN
VELOCITY
ℓ_1
ℓ_2
d_1
d_2
FLEXURAL MODE
$\left(\frac{\ell_1}{3.79}\right)^2 = \frac{K}{\omega}\sqrt{\frac{E}{\rho}}$
$\left(\frac{\ell_2}{2.47}\right)^2 = \frac{d_2}{d_1}\frac{K}{\omega}\sqrt{\frac{E}{\rho}}$
$(d_1/d_2)^{3/2} \approx$ VELOCITY TRANSFORMATION
(b)
STRAIN
VELOCITY

FIG. 2.—Design Data for Velocity Transformers Used in Ultrasonic Testing.

Typical velocity transformers consist of metal stubs, of varying section, rigidly bonded to the transducer face. An increase in particle motion is obtained at the expense of decreasing the area of the work face. Three types of resonant transformer have been found particularly useful in high power ultrasonics work; linear and exponentially tapered half-wavelength $\frac{\lambda}{2}$ stubs and double $\frac{\lambda}{4}$ cylindrical stubs. Their design features have recently been discussed by the author (12) and by Balamuth (13) and Merkulov (14). Double-$\frac{\lambda}{4}$ transformers give the largest transformation ratio for a fixed ratio of end diameters. They are also easier and cheaper to make and are the only type we need consider for this application. These are $\frac{\lambda}{2}$ resonant stubs composed of two cylindrical $\frac{\lambda}{4}$ sections of unequal area, as shown in Fig. 2. In the extensional or torsional mode, the distribution of velocity and stress in each section is sinusoidal, and it is easily seen that the peak velocity and stress in the small-diameter section are both greater than in the wide section by the ratio of



$$\ell = \left(\frac{2c'}{\omega}\right) \tan^{-1} \frac{2\omega}{\gamma c'}$$

Where $\gamma = \frac{4}{\ell} \log_e \frac{d_2}{d_1}$

$$C' = C\left(1 - \frac{\gamma^2 C^2}{4\omega^2}\right)^{-\frac{1}{2}}$$

C = Velocity of Sound in Material

ω = Resonant Angular Frequency

$\frac{d_1}{d_2}$ = Stress Amplification (G)

$$\left(\frac{d_1}{d_2}\right)^2 = \cot k\ell_1 \cot k\ell_2$$

Where $k = \frac{\omega}{C} = \frac{2\pi}{\lambda}$

λ = Vibration Wavelength

$\frac{\cos k\ell_1}{\sin k\ell_2}$ = Stress Amplification (G)

FIG. 3.—Metal Fatigue Test Specimens.

the areas of the sections. The theoretical distributions of particle velocity and strain in stubs for transforming extensional or torsional vibrations are indicated in Fig. 2(*a*). The length of each $\frac{\lambda}{4}$ section is $\frac{\pi c}{2\omega}$, where c is the velocity of longitudinal or transverse vibrations in the rod. Transformation of velocity in flexing stubs is also easily achieved by area reduction, preferably by stepping at a strain antinode, as suggested in Fig. 2(*b*). In this case, the small-diameter section resonates as a built-in cantilever with vibrating support.

Fatigue strength and acoustic loss are the chief factors determining the choice of material for a transformer. An ideal material should also be nonmagnetic, hard, and easily machined. Of readily available metals, the best are K-monel, various aluminum bronzes including Hidurax, certain light alloys, and titanium alloys (for example titanium 318A). A flange turned on the transformer in the plane of its central displacement node can be used for mounting the vibrator. It is essential to use a rigid flange. Some damping is always introduced here due to lateral strains introduced by Poisson's ratio effects. Screwed joints may be used between the transducer, velocity transformer, and fatigue specimen. A spot of grease or a thin soft copper washer may be used to provide good mating of the surfaces. But little stress is required to be transmitted at these junctions, which are always near the stress nodes.

DESIGN OF SPECIMENS

To avoid overstraining the drive vibrator, some stress amplification should occur in the specimen itself, which is therefore designed as a step-up stress transformer. Specimens may be symmetrically tapered or of dumbbell form as shown in Fig. 3. The simplest method of increasing the stress is to establish an area discontinuity at some point in the specimen, as in Fig. 3(*b*). The stress rise is then simply the area ratio. Consider, for example, operation in longitudinal push-pull. The distribution of oscillatory amplitude, ξ, from O to A (Fig. 3(*b*)) is

$$\xi = \xi_o \cos kx$$

where:

$k = \frac{2\pi}{\lambda} = \frac{\omega}{c},$

λ = vibration wavelength,

c = velocity of sound in specimen material, and

ω = angular frequency.

The strain,

$$\frac{\partial \xi}{\partial x} = -k\xi_o \sin kx;$$

$$\left(\frac{\partial \xi}{\partial x}\right)_A = -k\xi_o \sin kl_1.$$

From O' to A, $\xi = \xi_x \sin kx$, where ξ_x must be obtained from the boundary conditions at A.

$$\frac{\partial \xi}{\partial x} = k\xi_x \cos kx$$

$$\left(\frac{\partial \xi}{\partial x}\right)_A = k\xi_x \cos kl_2$$

The boundary conditions at A give:

$$(1) \qquad \overrightarrow{\xi_A} = \overleftarrow{\xi_A}; \quad \xi_x = \xi_o \frac{\cos kl_1}{\sin kl_2}$$

$$(2) \qquad d_1^2 \overrightarrow{\left(\frac{\partial \xi}{\partial_x}\right)_A} = d_2^2 \overleftarrow{\left(\frac{\partial \xi}{\partial_x}\right)_A}$$

Thus:

$$R = \left(\frac{d_1}{d_2}\right)^2 = \cot kl_1 \cot kl_2 \ldots\ldots (1)$$

where d_1, d_2, l_1, and l_2 are as shown in Fig. 3(*b*).

Equation 1 is the chief design equation. The theoretical distribution of oscillatory motion and strain in a transformer of diameter ratio 3:1 is given in Fig. 3(*b*). Transformers of this sort have the disadvantage that stress concentrations above those calculated from the area ratio are produced at the discontinuity unless corners are carefully filleted. But they have the advantage that the stress amplification can be made large. The central region is also quite uniformly stressed, the stress being more uniform the shorter the constricted region. The maximum strain is

$$\left(\frac{\partial \xi}{\partial x}\right)_{o'} = k\xi_x = k\xi_o \frac{\cos kl_1}{\sin kl_2}$$

The maximum strain obtained in a parallel $\frac{\lambda}{2}$ stub with end amplitude ξ_o is $k\xi_o$ so that the strain gain resulting from the area reduction is

$$G = \frac{\cos kl_1}{\sin kl_2} \qquad (2)$$

Figures 4(*a*) and (*b*) show theoretical curves plotted from Eqs 1 and 2. These curves, which relate l_1, l_2, and R; and G, l_2, and R, include all the information necessary for design purposes.

In designing stress transformers some preliminary measurements are often necessary to decide the minimum radius required at the area discontinuity to reduce unwanted stress concentration. Satisfactory shapes of specimens which have actually been used at 20 kcps are indicated in Fig. 3(*c*) and (*d*). A strain ratio of at least 2.5:1 should be chosen to ensure linear operation of the drive system and to keep the drive-face amplitude low enough to allow screw attachments. In designing for different materials at the same frequency, all longitudinal dimensions are proportional to the velocity of sound in the material.

None of the specimen designs discussed above allows uniform stressing of a large volume of material. Where this is essential, a special shape is required. It can be shown that the condition of uniform stress over a finite length demands a smoothly tapered profile which follows a gaussian curve (of the form $d = d_1 e^{\alpha x^2}$). In this case it is difficult to predict theoretically the resonant length of specimens, and empirical tuning is preferable.

When testing materials with high damping, such as certain steels, nimonics, etc., either external cooling must be applied or the specimen must be designed for efficient conduction of heat away from the test region (see, for example, the specimen of Fig. 3(*d*)). This condition implies restricting the volume of highly stressed material and requires a profile that results in a rapid variation of stress. When testing a large volume of such material, liquid cooling is essential. The specimen may be bored axially to carry a liquid coolant (Fig. 3(*e*)) or, preferably, a liquid spray may be applied to the outer surface, as indicated in Fig. 3(*f*). The heat extracted by the liquid may be used to measure the damping loss in the specimen.

For testing a material available only in small bulk (for example, rare or expensive metals) the specimen may be designed as a composite structure, as indicated in Fig. 3(*g*) where only the central, tapered, stress-transforming portion is of the test material. The size of this portion can be reduced to any convenient value and the dimensions of the end pieces adjusted to give the correct resonant frequency.

We have considered in some detail the design of specimens for axial push-pull tests. Designs are similar for torsion if the appropriate frequency constant for this mode is used. Symmetrical stepped stubs are also satisfactory for tests in flexure, but in this case it is difficult to predict the resonant dimensions and stress transformation theoretically, and an empirical approach is preferable.

THE DRIVE GENERATOR

The conventional method of driving electromechanical transducers is to use an electronic oscillator-amplifier matched to the transducer. Apart from the high-frequency drive, polarization is required. For piezoelectric ceramics, this is obtained by operation about remanence, but for piezomagnetic transducers it is often preferable to provide continuous polarization by passing a direct current through the drive coil. Suitable connections for driving in axial push-pull or torsion are shown in Fig. 5(*a*). For exciting flexural vibrations, the two opposing limbs of the transducer must be driven in opposite phases. This can be arranged by supplying polarization and drive in either of the two ways shown in Fig. 5(*b*) and (*c*).

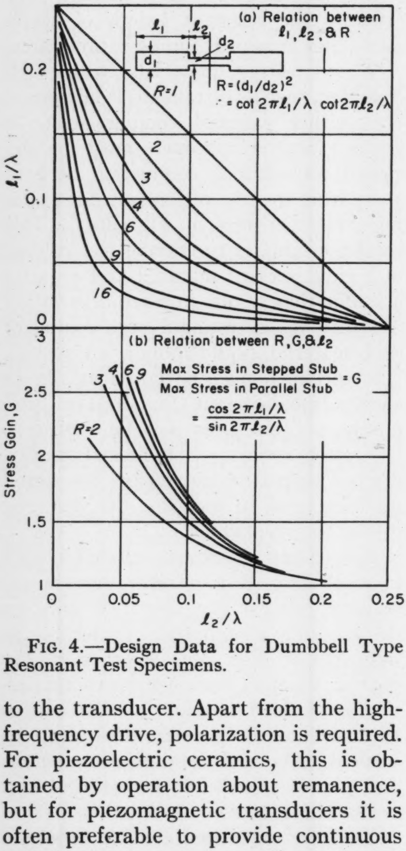


FIG. 4.—Design Data for Dumbbell Type Resonant Test Specimens.

The drive generator supplies the power dissipated by mechanical hysteresis in the fatigue specimen. In practice this is a quite variable quantity and depends on: (1) The type of material used; (2) the stress level used in the test (the *Q* decreases very rapidly as the stress increases towards the fatigue limit); (3) the volume of material tested; and (4) the distribution of stress in the specimen. Items (3) and (4) are fixed by the geometry of the specimen and may be standardized for the equipment. Then for any material, only the loss-strain variations need be considered in relation to acoustic matching. For maximum efficiency, the drive vibrator should be made to operate into its optimum acoustic load (12). The best condition is obtained when this is identified with the maximum load obtained during tests (that is, that encountered near the fatigue limit). The value of velocity transformation required can be estimated as explained in reference (12). For efficient operation, therefore, acoustic matching is necessary whenever the specimen material or design is changed. For a versatile tester it is obviously more economical to tolerate some inefficiency in power transfer, thereby avoiding a lengthy design procedure; operation at a low over-all efficiency has the practical advantage that a wide range of loads can be used without changing the matching to the drive vibrator or amplifier. There is the disadvantage that the loaded *Q* of the system may be high, making manual tuning inconvenient. But simple, self-regulated systems have been used very successfully. For example, a voltage proportional to the oscillatory motion of the transducer can be obtained using either a piezoelectric crystal bonded to the vibrator, or a charged capacitor plate supported close to the transducer face. The output from the pickup is taken to the amplifier input *via* preamplifier, phase shift, and limiter circuits, as indicated in Fig. 5(*d*).

Mason (15) has discussed pulsed methods of driving ultrasonic fatigue test vibrators. A pulsed drive avoids overheating of the specimen, which is always a problem at the higher frequencies. But the fatigue limits under pulsed and continuous-drive conditions differ, and test conditions are therefore complicated. If adequate cooling can be achieved by other means it is better to avoid pulsing.

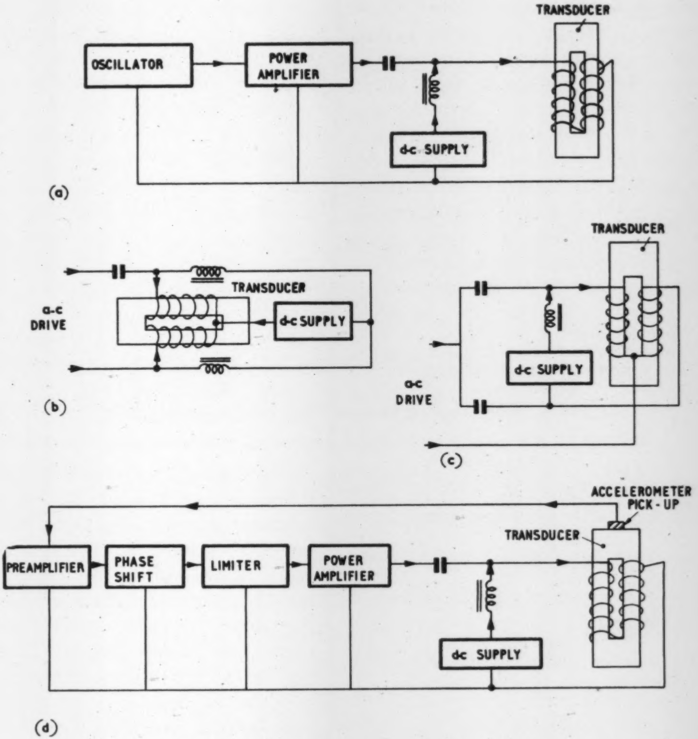
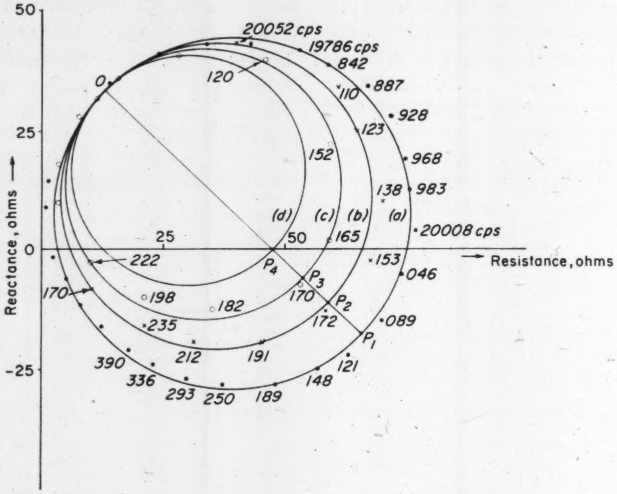

TRANSDUCER
OSCILLATOR
POWER AMPLIFIER
dc SUPPLY
(a)
TRANSDUCER
a-c DRIVE
TRANSDUCER
d-c SUPPLY
(b)
a-c DRIVE
d-c SUPPLY
(c)
ACCELEROMETER PICK-UP
TRANSDUCER
PREAMPLIFIER
PHASE SHIFT
LIMITER
POWER AMPLIFIER
dc SUPPLY
(d)

FIG. 5.—Drive Generators for Piezomagnetic Vibrators.

THE MEASURING SYSTEM

The chief quantities of interest in fatigue studies are: (1) peak and mean strain; (2) dynamic elastic modulus; (3) elastic damping; and (4) the number of cycles of strain.

The performance of the vibrator can be assessed by electrical impedance or admittance measurements taken at the transducer terminals and by measurements of oscillatory motion recorded microscopically or by using any convenient type of vibration pickup. Plots of velocity and strain obtained in this way for the useful types of mechanical transformers are given in Figs. 2 and 3. Fig-



(*a*) Free transducer.
(*b*) Transducer with velocity transformer.
(*c*) Transducer with velocity transformer and specimen (low strain).
(*d*) Same as (*c*) at high strain.

FIG. 6.—Electrical Impedance Plots for Ultrasonic Tester.

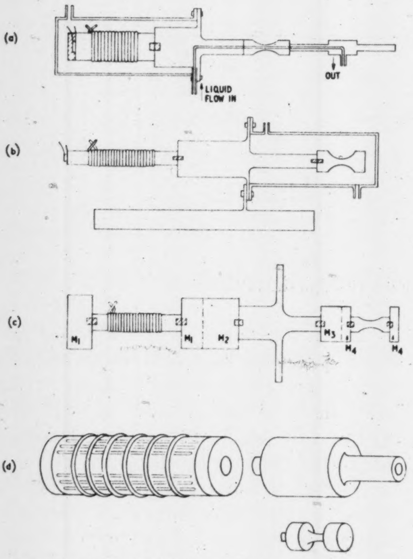


FIG. 7.—Practical Ultrasonic Test Vibrators.

ure 6 records a series of impedance plots taken during the development of the vibrator shown in Fig. 7(*b*). Addition of low-loss velocity transformers causes small reductions in the diameter of the impedance circle and an increased spacing of the frequency points around the circle. The former gives an indication of the added damping and the latter is a measure of increased *Q* of the system (compare (*a*) and (*b*), Fig. 6). Addition of a resonant fatigue specimen causes a further increase in damping ((*c*), Fig. 6).

Of particular interest in fatigue work are the changes that occur in elastic dissipation and dynamic modulus with strain level and time. These should preferably be recorded automatically. An electronic impedance tracer has therefore been used (16). The Argand diagram is traced on a cathode-ray-tube screen, if necessary many times per minute. A permanent record can be obtained photographically. Circle (*d*), Fig. 6, illustrates the reduction in circle diameter obtained when the specimen is subjected to stress in the nonlinear region. In Fig. 6 the impedance circles all touch at the common point O, which refers to the clamped electrical impedance of the system. The diameters through O (motional diameters) lie on the line OP cutting the circles at the frequency points of mechanical resonance (P_1 to P_4). The motional diameter, D, is inversely proportional to the total mechanical resistance of the system; thus, for small changes the increased loss, $+\Delta R$, or decreased Q, $-\Delta Q$, which follows the strain increase in the specimen, is obtained. At the same time, any change in the elastic modulus is reflected in a change in the resonant frequency of the system, that is, a change in the frequency of the point P on the circle. The frequency difference, $\Delta\omega$, is proportional to the change ΔE_1, in the real part, E_1, of the complex modulus. Thus, the changes in the complete complex dynamic modulus, $E = E_1 + jE_2 = E_1\left(1 - \frac{j}{Q}\right)$, are obtained. Because of the strong dependence of ΔE_1 and ΔQ on the strain as the fatigue limit is approached, it is essential to plot out the impedance loci at constant strain, at least for all frequencies near resonance.

At ultrasonic frequencies the total elastic dissipation is great enough to allow calorimetric methods to be used for its measurement as an alternative to impedance plots. This method is discussed later. It is more direct, but cannot be used to reflect rapid changes in damping.

The most direct method of measuring strain is to plot the oscillatory amplitude, ξ, along the specimen, obtained with a microscope. The strain distribution is obtained from the slope of the (ξ, x) curve. Thereafter, a single amplitude measurement gives the peak strain. This can be made with a microscope but it is better to use an accelerometer consisting of a small disk of prepolarized barium titanate or lead zirconate-titanate stuck on near a displacement antinode of the system; the voltage output is measured or recorded. The same accelerometer can be used for self-regulation of the vibrator, part of the output being fed back through phasing and limiter circuits to the amplifier input. The limiter control is then also the strain level monitor. If a barium titanate transducer is used for driving the vibrator a separate pickup crystal is superfluous (17). An advantage of the self-regulated system is that a higher operating Q is tolerable.

The number of cycles of stress can be recorded automatically by a mechanical or electronic counter.

PRACTICAL FATIGUE TEST SYSTEMS

Figure 7(*a*) shows the vibrator for a medium-power tester used to study damping in relation to fatigue. The vibrator is designed for one frequency only, 20 kcps, and operates in free-free tension-compression. The mechanical line consists of four $\frac{\lambda}{2}$ resonant sections. A single screw attachment is used as coupling between the laminated nickel transducer and the 6:1, double-$\frac{\lambda}{4}$ velocity transformer, the mating faces of which are ground accurately flat and tightened up on a thin soft copper washer to ensure a perfect acoustic bond. The fatigue specimen is cooled by an axial flow of liquid which enters the system on the low-stress side of the displacement node of the velocity transformer. The sole purpose of the final $\frac{\lambda}{2}$ section is to carry the cooling liquid outflow. It is a 4:1 step-down stress transformer and the liquid emerges at the displacement node, where also the alternating stress is small enough to avoid fatigue of the outflow pipe connections. Couplings between velocity transformer, specimen, and outflow stub are also made by screw threads, the stress ratio of the specimen being large enough to allow this. The use of an axial flow path complicates the design of the specimen, but at 20 kcps these are so short that a 3-mm-diameter hole can be bored rapidly and accurately and the surface finished to any required specification with little difficulty. The vibrator can be supported on a flange turned on the velocity transformer in the plane of its displacement node. It may be necessary to water-cool the transducer; the nodal flange then serves as a support for the water jacket. If liquid cooling is used, the free radiating surfaces of transducer and transformer must be protected from radiation loss and erosion damage by fixing acoustic pressure release material, for example, sponge rubber, as indicated in Fig. 7(*a*). A small barium titanate accelerometer attached to the transducer face is used to monitor the strain and to provide a voltage signal for self-regulation.

The axial flow, in addition to cooling the specimen, can be used as a means of measuring elastic dissipation. Thermocouples have been used for measuring the difference temperature between inflow and outflow. Both the velocity transformer and outflow stubs on either side of the specimen are made of a low-oss alloy (for example Hiduminium 77) to minimize the loss correction necessary. At high strains most of the acoustic dissipation occurs in the central, reduced portion of the specimen, which is designed for constant strain over this region; the measured dissipation then relates to a definite measured strain.

The use of liquid cooling complicates test conditions, and for some materials there is danger that chemical attack or corrosion may limit the fatigue life. The alternatives are to use forced air or to avoid external cooling altogether by designing the specimen to give rapid conduction of heat away from the highly stressed central region (for example, see specimen Fig. 3(*d*)). A multijet air blast has been found satisfactory for many metals (see Fig. 3(*f*)). Figures 7(*b*) and 8 show the vibrator for a low-power (60-w) tester using a specimen which may be cooled either by forced air or

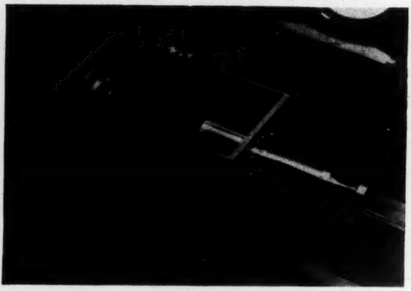


FIG. 8.—60-w, Free-Free Axial Load Tester.

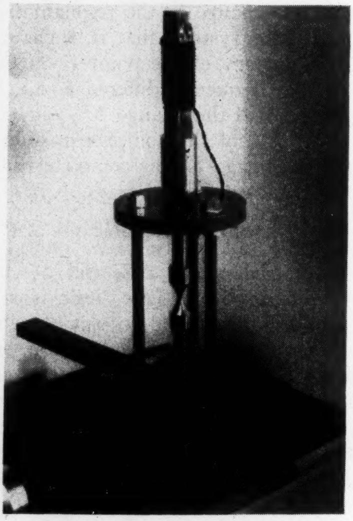


FIG. 9.—60-w Tester for Tests with Steady Tension.

heat conduction. The vibrator is a three-$\frac{\lambda}{2}$ system, using a small laminated-nickel transducer with screw-on, double $\frac{\lambda}{4}$, 7:1 velocity transformer and a dumbbell-type specimen. It is intended for single-frequency testing in free-free tension-compression. Many of the constructional features are similar to the previous system. This vibrator design is ideal for testing under varying environmental conditions—the flanged nodal support can be used to carry a sealed jacket, surrounding the specimen, which can be filled with the appropriate gas at the required temperature and pressure.

Figure 9 shows a method of mounting the vibrator for testing in push-pull under a superimposed steady tension or compression. The vibrator is identical with that of Fig. 8 except that the nodal support flange is omitted and an extra $\frac{\lambda}{2}$ parallel resonant section is required. The vibrator is fixed in a simple frame with two supports, the lower of which carries a lever pinned to the central displacement node of the final stub. The specimen is loaded by adding weights to the lever arm. Screwed joints have been found to couple very efficiently under steady tensions up to at least 80,000 psi. Clamping the vibrator in two planes accentuates the need for accurate tuning of the separate sections of the line. Unless the supporting planes are accurately nodal, additional loss is incurred when the tension is applied. In practice, power requirements are found to be about 4 to 5 times greater than for the free-free system. About 250 w drive power is required and some external cooling of the vibrator is essential.

The above systems are resonant devices suitable for testing at only one frequency. We have found that ultrasonic vibrators may also be designed to resonate in a very wide range of frequencies, extending down to overlap those of conventional testers. This is made possible by providing a series of screw-on masses which can be added or subtracted, as required, from the separate sections of the vibrator. Addition of lumped mass M_1 to a vibrator of equivalent mass M^* reduces the resonant frequency in the ratio $\left(\frac{M^*}{M^*+M_1}\right)^{1/2}$. Fatigue test vibrators can thus be operated at a number of spot frequencies covering any required range. Such a system is shown in Fig. 7(*c*). A variable high-frequency tester of this sort has great potential value both in industry and research.

The vibrators described above all operate in axial push-pull. This is the most useful of the available modes for fatigue work. Ultrasonic transducers are also available if tests are required in torsion or bending, and a single transducer can be designed for use in all three modes; Fig. 7(*d*) shows a piezomagnetic vibrator of this type. The transducer is a laminated scroll carrying five windings which, by suitable connections of drive and polarization currents can be made to resonate in longitudinal push-pull, torsion, or flexure in either of two perpendicular directions. The end faces carry two metal disks, one of which is tapped to receive the velocity transformer. The other carries a barium titanate accelerometer for monitoring the strain and providing a signal for self-regulation. The complete vibrator is a three-$\frac{\lambda}{2}$ system; in changing the mode of test, both the velocity transformer and specimen must be changed even if the dimensions of the transducer are so chosen that it resonates at the same frequency in all modes. A piezomagnetic tube can also be used.

Finally, the St. Clair type generator has been used successfully in high fre-

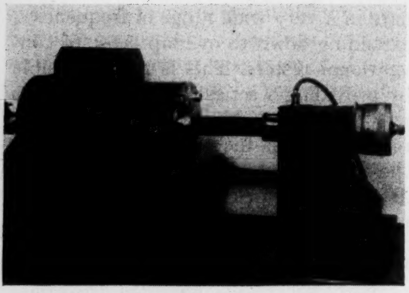


FIG. 10.—Ultrasonic Tester Based on Electrodynamic Transducer.

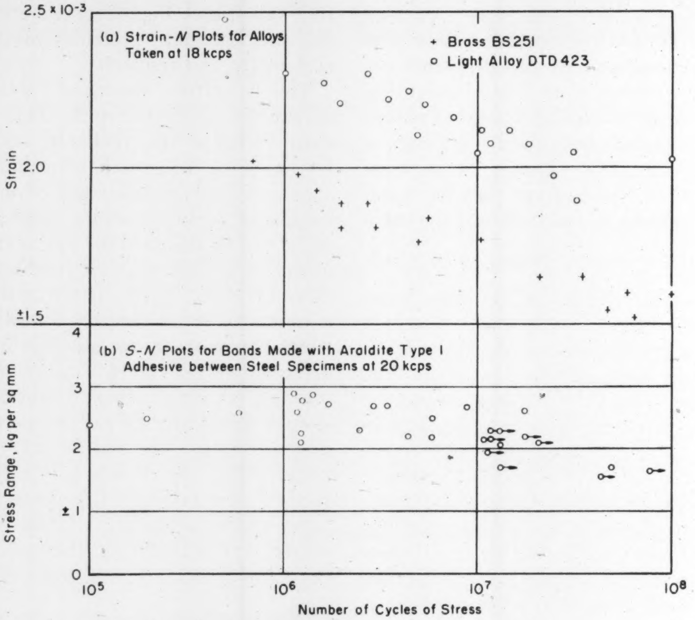


FIG. 11.—Fatigue Results for Metals and Adhesives.

quency fatigue work. Since these vibrators are driven electrodynamically, any low-loss, high-strength metal can be used for the resonant member. If a very-high-strength alloy such as titanium 318 or Hiduminium 77 is used, then for testing many materials it may not be necessary to provide any stress transformation in the fatigue specimen. This can therefore be a simple $\frac{\lambda}{2}$ parallel-sided section, bolted directly to the vibrator face, as in Fig. 10. This advantage of the electrodynamic transducer in allowing a simple design of specimen is to some extent offset by the inconvenience of a large vibrator which is rather expensive to build and difficult to set up. It is also capable of operation in only one mode—axial push-pull.

PERFORMANCE AND TEST RESULTS

The instruments described above have been widely used in tests on steels, light alloys, bronzes and brasses. Specimen results are given in Figs. 11 and 12.

Figure 11(*a*) shows plots of strain *versus* number of cycles, *N*, for axial push-pull at 18 kcps for extruded brass (BS-251), and for a typical aluminum alloy (DTD 423 extruded rod). Similar plots have been obtained for many other light alloys, bronzes and ferrous alloys. Fatigue limits are up to 40 per cent higher than for low frequencies. It will be interesting to discover how the fatigue limit varies with frequency over the range covered by the variable frequency tester described above.

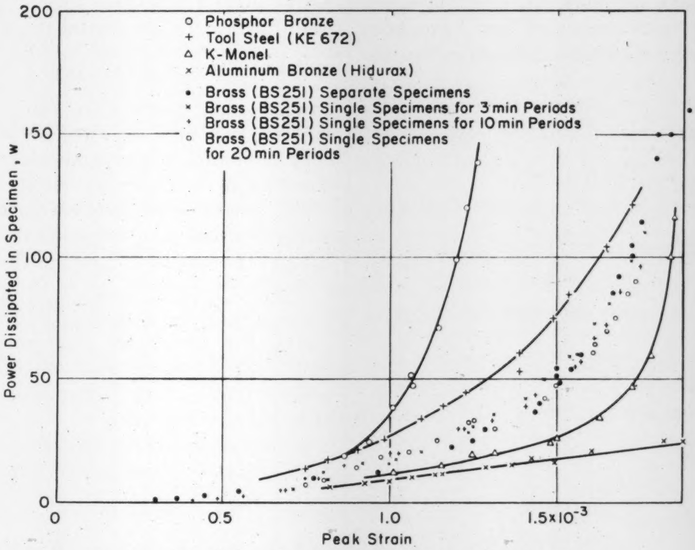


FIG. 12.—Loss-Strain Characteristics for Various Alloys at 18 kcps.

The start of a fatigue crack is always accompanied by a sudden change in the electrical impedance recorded on the circle tracer. When the crack has propagated a short distance, stress is so reduced by acoustic mismatch that further progress is halted. Since the crack occurs at a displacement node, a broken specimen becomes a $\frac{\lambda}{4}$ section, clamping the system, and the mechanical impedance increases sharply. Examination of broken specimens has shown that the crack invariably starts at the outer surface of the waist region and proceeds inward, even when the specimen contains an axial cooling hole.

Many measurements have been made of acoustic loss as a function of the number of cycles of stress and of the applied stress, using the techniques described above. The power loss-strain characteristics of some of the alloys tested are given in Fig. 12. Little variation of loss with time could be observed for these materials even at stresses near the fatigue limits. But it must be remembered that at the test frequency, 20 kcps, the changes occurring during the first or last 10^4 cycles cannot be distinguished, and rapid changes may well be taking place during these periods.

The curves of Fig. 12 show how the acoustic loss increases rapidly with strain as the fatigue limit is approached.

This increase is the result of a rapid broadening of the elastic hysteresis loop in the nonlinear stress-strain region. The accompanying decrease in the real part of the elastic modulus has also been recorded. The loss-strain characteristic can evidently be used as an alternative to strain *versus* N plots to indicate the fatigue range. It may even be possible to infer the fatigue limit from loss-strain measurements made on a single specimen. Measurements of loss have been obtained by increasing the stress to the fatigue limit in an equal number of small steps, but varying the lengths of the individual stressed periods. The loss-strain characteristics obtained in this way are compared with each other and with the plots for separate specimens. Measurements show a definite trend toward increase of fatigue limit with increase in the length of the "understressed" periods (increased pulse length). The loss-strain characteristic obtained for the shortest pulse length appears indistinguishable from that for separate specimens. The fatigue limit may then be deduced from measurements on a single specimen. Testing is thus greatly accelerated, and a fatigue limit obtained in a few minutes. Figure 12 includes loss-strain plots for brass specimens tested in this way.

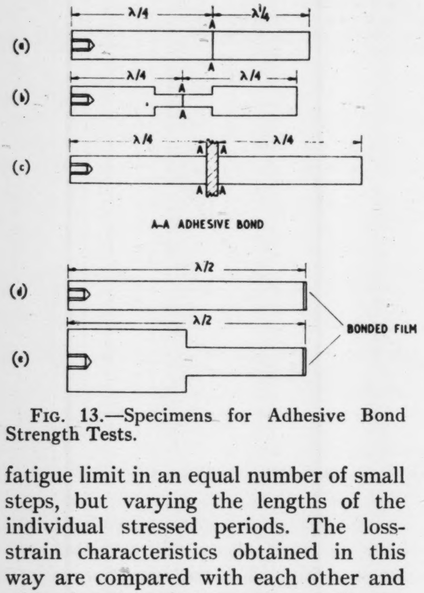


FIG. 13.—Specimens for Adhesive Bond Strength Tests.

The ultrasonic equipment is useful not only for testing metals. Provided the acoustic loss is not too great, tests can be made on nonmetals such as plastics, glass, and ceramics. In particular, the technique is easily applied to testing adhesive bond strength.

1. For bonds of metal-to-metal or metal-to-low-loss solid, two $\frac{\lambda}{4}$ straight parallel-sided pieces of material of the same section may be used, butt-jointed over flat surfaces perpendicular to the direction of vibration as shown in Fig. 13(*a*). The distribution of oscillatory motion and stress along the stubs is sinusoidal and the maximum stress at the center is $\frac{2\pi E\xi}{\lambda}$, where E is Young's modulus and ξ the maximum peak oscillatory amplitude. Thus the peak stress at the bond is referred to a single measurement of oscillatory amplitude.

2. When the bond strength is very high, to avoid inconveniently large drive powers, the $\frac{\lambda}{4}$ sections can be stepped and the bond made between the small-area portions (Fig. 13(*b*)).

3. Where one material is in sheet form, the best method is to bond the sheet between $\frac{\lambda}{4}$ rod sections of the required second material (Fig. 13(*c*)). In this way two bonds are tested together, the peak stress at the bond being $\frac{2\pi E\xi}{\lambda}$, where ξ is the peak oscillatory amplitude in either $\frac{\lambda}{4}$ stub, and E is Young's modulus for the stub material. It is important to ensure that the area of the sheet is greater than that of the stub. This avoids

undesirable stress amplification across the bond.

Bonding tests have been made using a wide range of well known, high-strength adhesives. Tests were made on steel-to-steel and steel-to-ferrite bonds, the latter being of interest in view of the recent development of ferrites for ultrasonic work. For both types of bond, the dynamic strength was found to be greatest for hot setting Araldite, for which the strength was at least twice that of the others. Specimen results for steel-to-steel bonds are presented in the form of *S-N* plots in Fig. 11(*b*).

Measurement of the adhesion strength of thin films is a further valuable application of the ultrasonic test gear. In Figs. 13(*d*) and (*e*), consider the alternating stress developed at the bonded surface of a film of material of thickness t, and density ρ, adhering to the end surface of a $\frac{\lambda}{2}$ longitudinal vibrator, angular frequency ω, driven at peak oscillatory amplitude ξ. We assume that t is much less than λ in the film material. The peak stress developed at the bond is $\omega^2\xi\rho t$. For a given film thickness the maximum stress obtainable is proportional to the peak particle acceleration, $\omega^2\xi$, produced at the free-end face of the vibrator. This is rather different from normal requirements for fatigue testing; here there is no question of stress transformation and the aim is to achieve the maximum possible particle motion. The maximum velocity is fixed by the fatigue strength, F, and characteristic mechanical impedance, ρc, of the vibrator material. For a cylindrical $\frac{\lambda}{2}$ or double-$\frac{\lambda}{4}$ stub, $(\omega\xi)_{\max} = \frac{F}{\rho c}$. The stub material should therefore be chosen for maximum value of this parameter.

For testing materials which have high damping or nonlinear stress-strain behavior, it is not convenient to use resonant specimens. A small disk specimen can be used, pressed into contact with the vibrator drive face. The other face of the specimen should be clamped, using a $\frac{\lambda}{4}$ stub which is an infinite load at the drive frequency. The arrangement is similar to Nolle's equipment for measuring the complex modulus of rubber-like materials (18). Measurements of the change in resonant frequency and mechanical Q due to the specimen loading allow the real and imaginary parts of the dynamic modulus to be obtained. In this application it is advisable to use a transducer with low electrical loss (for example, piezomagnetic ferrite) to avoid asymmetry in the electromechanical response. Low frequency testing can be done by using mass-loaded systems, as already indicated. The nonlinear behavior of complex materials such as plastics and rubbers, as well as metals with high damping, can in this way be studied.

Finally, these high energy ultrasonic vibrators have been found useful for accelerated testing of wear produced either by friction or hammer blows. Mason (17) has discussed this application. Vibrators are used with suitable end probes or tools pressed into contact with the specimen with the required steady pressure. An important extension of this technique is ultrasonic machining, where the hammer-action principle is used for drilling shaped holes and for lapping and polishing by rubbing action (12).

SUMMARY—ADVANTAGES AND LIMITATIONS

These vibrators open up a new field of fatigue testing. They allow testing at frequencies from a few thousand up to at least 50 thousand cps. The increased speed is a great advantage in long-duration tests of 10^8 cycles or more. Tests with conventional machines are practically restricted to a maximum of about 10^8 cycles. Ultrasonic vibrators allow an extension to at least 10^9 and probably 10^{10} cycles. This will be particularly valuable in research. On the other hand, these testers cannot conveniently be used to investigate fatigue behavior over the whole of the specimen life; it will be necessary to revert to a low frequency to cover the first 10^4 to 10^5 cycles.

Measurements have confirmed that the fatigue limit is a function of the operating frequency. This complicates testing, and where strength data for low-frequency service use are required, the ultrasonic tester will need to be calibrated relative to the low frequencies experienced by the component in service. A valuable research contribution will be to use ultrasonic-type vibrators to investigate fully fatigue behavior over the range 1 to 50 kcps, and this work is being planned.

The simplicity of the high-frequency vibrator is worth emphasizing. The apparatus contains no rotating or macroscopically-moving parts. Even with facilities for variable-frequency, steady stress, and automatic recording the equipment is still simpler and less expensive than conventional testers. Facilities can readily be included for testing in any or all of the three useful modes of vibration. The equipment is also easily adapted to axial push-pull tests with steady tension or compression, and variable-frequency operation. Specimens are of simple form and require little material. Easy replacement is ensured by screw attachment. Specimens designed for constant stress distribution allow tests to be done on a large volume of material, if required. Tests can be performed under variable temperature or pressure conditions, or in a corrosive atmosphere. This is a particularly useful feature, since more importance is now being attached to environmental conditions of test, and many conventional machines cannot easily be adapted to such tests.

A disadvantage of ultrasonic testing is the increased elastic dissipation obtained at the higher frequencies. But by suitable specimen geometry, the need for external cooling can often be avoided.

The ultrasonic equipment is applicable to a wide range of tests on metals, glass, ceramics and plastics, including adhesive strength tests and tests on the bonding strength of films and protective coatings on metals. The measuring system is sufficiently sensitive to detect the formation of incipient cracks in specimens.

REFERENCES

(1) C. F. Jenkin, "High Frequency Fatigue Tests," *Proceedings*, Royal Soc. (London), A, Vol. 109, p. 119 (1925).

(2) C. F. Jenkin and G. D. Lehman, "High Frequency Fatigue," *Proceedings*, Royal Soc. (London), A, Vol. 125, p. 83 (1929).

(3) R. F. Hanstock and A. Murray, "Damping Capacity and the Fatigue of Metals," *Journal of the Institute of Metals* (London), Vol. 72, p. 97 (1946).

(4) N. Thompson, N. Wadsworth, and N. Louat, "The Origin of Fatigue Fracture in Copper," *Philosophical Magazine*, Serial 8, Vol. 1, p. 113 (1956).

(5) T. W. Lomas, J. O. Ward, J. R. Rait, and E. W. Colbeck, "The Influence of Frequency of Vibration on the Endurance Limit of Ferrous Alloys at Speeds up to 150,000 Cycles per Minute Using a Pneumatic Resonance System," *Proceedings*, Int. Conference on Fatigue of Metals, Institution Mechanical Engrs. (London) and Am. Soc. Mechanical Engrs. (1956).

(6) A. R. Wade and P. Grootenhuis, "Very High Speed Fatigue Testing," *Proceedings*, Int. Conference on Fatigue of Metals, Institution Mechanical Engrs. (London) and Am. Soc. Mechanical Engrs., *Paper No. 5* (1956).

(7) W. P. Mason, "Piezoelectric Crystals and

their Application in Ultrasonics," Van Nostrand Co., Inc., New York, N. Y., p. 161 (1950).

(8) E. A. Neppiras, "Metal Fatigue at High Frequency," *Proceedings*, Physical Soc., B (London), Vol. 70, p. 393 (1957).

(9) W. P. Mason, "Electromechanical Transducers and Wave Filters," Van Nostrand Co., Inc., New York, N. Y. (1956).

(10) T. F. Hueter and R. H. Bolt, "Sonics," John Wiley and Sons, Inc., New York, N. Y. (1955).

(11) H. St. Clair, "An Electromagnetic Sound Generator for Producing Intense High Frequency Sound," *Review of Scientific Instruments*, Vol. 12, p. 250 (1941).

(12) E. A. Neppiras, "Design of Ultrasonic Machine Tools," *Proceedings*, Conference on Technology of Engineering Manufacture, Institution Mechanical Engrs. (London), *Paper No. 6* (1958).

(13) L. Balamuth, "Mechanical Impedance Transformers in Relation to Ultrasonic Machining," *Transactions*, Inst. Radio Engrs. (Professional Group Ultrasonics Engineering), No. 2 (1955).

(14) L. G. Merkulov, "Design of Ultrasonic Concentrators," *Soviet Physics—Acoustics*, Vol. 3, p. 246 (1957).

(15) W. P. Mason, "Internal Friction and Fatigue in Metals at Large Strain Amplitudes," *Journal of the Acoustical Society of America*, Vol. 28, p. 1207 (1956).

(16) M. J. van Schagan, "Cathode Ray Display of Complex Quantities at Varying Frequencies," *Philips Technical Review*, Vol. 18, p. 380 (1956).

(17) W. P. Mason and R. E. Wick, "Barium Titanate Transducer Capable of a Large Amplitude at an Ultrasonic Frequency," *Journal of the Acoustical Society of America*, Vol. 25, p. 209 (1951).

(18) A. W. Nolle, "Methods for Measuring Dynamic Mechanical Properties of Rubber-Like Materials," *Journal of Applied Physics*, Vol. 19, p. 753 (1948).

DISCUSSION

MR. G. R. GOHN.[1]—This is one of a series of papers which has been offered in recent years on the subject and it is probably one that those engaged in fatigue testing will find most useful. The author not only gives the formulas for calculating strains and, if you wish, stresses, but also gives sufficient details so that any of the equipment described can be constructed. It is also the first experiment that I know of which has attempted to carry out these high-speed tests not only in axial loading but also in bending and torsion.

We too have been experimenting along these lines. Our own tests are being carried out at the moment at 17,400 cps, a frequency at which a large number of cycles can be built up in a very short period of time. We intend to follow this up in studying the relation between fatigue tests carried out in the conventional manner and fatigue tests carried out at ultrasonic speeds. This technique has also been found to be a very useful scheme for growing whiskers and studying the dislocation phenomena which are so evident in these small structures.

MR. BENJAMIN J. LAZAN.[2]—One of the difficulties in fatigue testing at very high speeds is the internal heating associated with the hysteresis damping in the material. Even if the specimen is surface cooled, either by a liquid or by air, there may be a serious temperature gradient from the surface to the interior. Does the paper include any data which indicate the magnitude of this gradient and its possible significance?

MR. E. A. NEPPIRAS (*author*).—I am interested to hear of the proposed work at Bell Laboratories. We seem to be working on similar lines and I will make a point of keeping Mr. Gohn informed of our future progress.

---

[1] Supervisor, Creep and Fatigue Laboratories, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] Professor of Materials Engineering, University of Minnesota, Minneapolis, Minn.

In reply to Mr. Lazan, I agree that generation of heat by hysteresis loss is a limitation to the use of very high frequencies for tension-compression testing. It will limit the volume of material that can be tested. No actual measurements of temperature gradients were made but it does seem clear that severe gradients would not be set up in the types of specimen used in the work reported.

A METHOD FOR DETERMINING THE FATIGUE LIMIT OF METALS BY MEANS OF STEPWISE LOAD INCREASE TEST*

BY N. ENOMOTO[1]

The Wöhler method is conventionally used for determining the fatigue limit of metals. In this, an *S-N* diagram is drawn, from which the fatigue limit may be determined. This method takes a long time and involves the testing of many specimens. Several accelerated testing methods have been proposed making use of the variation of physical properties in the process of fatigue. However, a reliable or practical method has not been established.

A method for predicting the fatigue limit by using a progressive load increase test was proposed by E. M. Prot (**1**).[2] The reliability of this method has been appraised by many investigators. In Prot's approach, it is assumed that there exists a linear relation between the stress at failure under linearly increasing load and the square root of the loading rate. Thus the stress obtained from this relation at zero loading rate is claimed to coincide with the Wöhler fatigue limit. The stress at failure, S_R, under progressive load increase, the loading rate, α, and the fatigue limit, S_e, are related by the following expression:

$$S_R = S_e + K\alpha^n, n = \tfrac{1}{2} \ldots\ldots \quad (1)$$

When this method is used, it is said that the testing time can be reduced as compared with the Wöhler procedure.

Upon evaluating the results of the Prot method, it was found that the value of n in Eq 1 was different for different materials. Further, the values obtained by several investigators were not in agreement and no generally accepted conclusion has been reached.

According to the author's experience, the Prot method was not considered particularly effective in saving time. In addition, the number of specimens required for testing by this method was not markedly fewer than by the Wöhler method. Furthermore, the progressive load increase test requires a special device to increase the alternating load linearly and existing machines cannot be employed without modification.

In the present paper a method is proposed for determining the fatigue limit by a fatigue test under stepwise increasing load with only one loading rate. The practicability of this method is appraised.

DETERMINATION OF FATIGUE LIMIT BY MEANS OF A PROGRESSIVE LOAD INCREASE TEST USING ONLY ONE LOADING RATE

The author (**2**, **3**), employing the idea of cumulative damage, has obtained the following relation between the *S-N* curve and progressive load increase fatigue test:

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Railway Technical Research Institute, Japanese National Railways, Tokyo, Japan.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

$$\alpha = \left[\frac{S_e}{N_c}\right] \left[\frac{1}{m+1}\left(\frac{S_R}{S_e}\right)^{m+1} - \frac{S_R}{S_e} + \frac{m}{m+1}\right] \quad (2)$$

where:

S_e is the value of fatigue limit,

TABLE I.—CHEMICAL COMPOSITION, MECHANICAL PROPERTIES AND HEAT TREATMENT.

	S10 Carbon Steel	S40 Carbon Steel
Chemical composition, per cent:		
Carbon	0.12	0.39
Silicon	0.28	0.12
Manganese	0.53	0.48
Phosphorus	0.034	0.031
Sulfur	0.017	0.040
Mechanical properties:		
Tensile strength, kg per sq mm	69.4	67.5
Yield strength, 0.2 per cent, kg per sq mm	...	39.9
Elongation, per cent	22.3	32.7
Reduction of area, per cent	47.4	55.0
Rockwell hardness, B scale	89.6	87.1
Heat treatment	760C, 20 min, water-quenched	850C, 30 min, normalized

N_c is the critical number of stress cycles,[3] and

$-1/m$ is the value of the slope of the finite region of the S-N curve on a log-log diagram.

For a progressive load increase fatigue test, α is the loading rate and S_R is the stress for rupture.

According to this equation, the stress for rupture becomes smaller and approaches the value of S_e as the loading rate is made smaller. However, in practice, if the loading rate is decreased beyond a certain limit, it has been shown that for some materials the stress for failure becomes larger, contrary to expectation. This indicates that understressing produces a strengthening effect. Accordingly, if a progressive load increase test is carried out using a loading rate somewhat larger than this limiting value, a failure stress close to the Wöhler fatigue limit will be obtained. The Wöhler fatigue limit may then be predicted if the difference between the Wöhler fatigue limit and rupture stress in a progressive loading test is known. According to many tests, this critical value of loading rate is fairly small, that is, of the order of 5 × 10^{-7} kg per sq mm per cycle.[4] An adequate loading rate can be maintained, using a fatigue testing machine running at ordinary cyclic speeds, by adding a small load increment at intervals of several hours. This procedure produces a uniformly increasing step-like stress history. If no appreciable difference is found between the strength for rupture obtained from the steplike load increase method and the linearly increasing stress method, the former method might be judged to have more value for practical use.

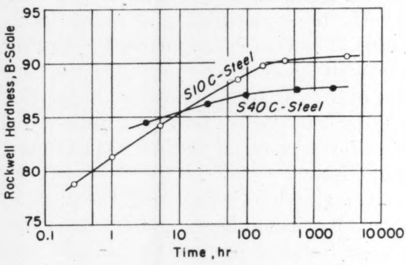


FIG. 1.—Change in Hardness of Specimens with Time After Heat Treatment.

Fatigue tests were carried out to de-

---

[3] This is indicated as the point of intersection of the line representing the finite and infinite region of the S-N curve.

[4] 1 kg per sq mm = 1422 psi.

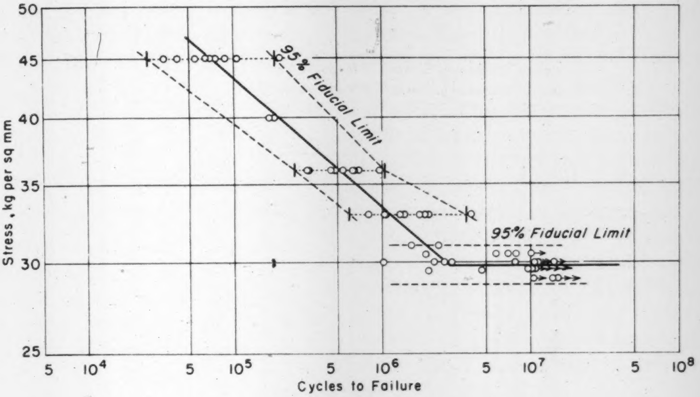


FIG. 2.—*S-N* Diagram for S 10 Carbon Steel.

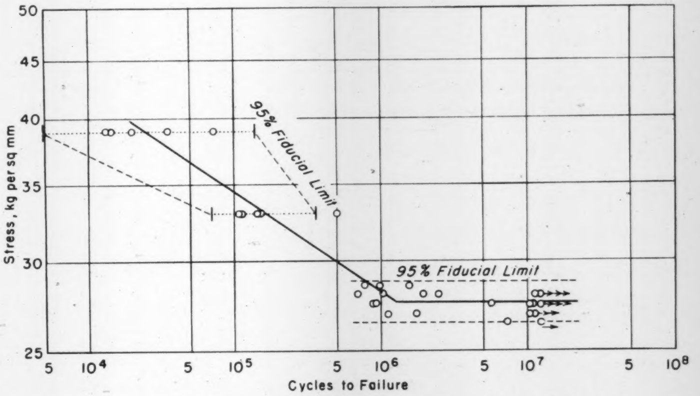


FIG. 3.—*S-N* Diagram for S 40 Carbon Steel.

termine the critical loading rate with water-quenched mild steel and normalized medium-carbon steel specimens which are supposed to be susceptible to strain aging. The chemical composisions and the mechanical properties of the test materials are listed in Table I. Figure 1 shows the progress of strain aging as measured by the hardness of the specimens after heat treatment; both curves show an increase of hardness with time indicating that strain aging does occur.

Rotating-beam fatigue machines running at a testing speed of 2100 rpm were used. The specimen diameters were 10 mm for S10 carbon steel and 9 mm for S40 carbon steel. Specimens were turned and ground after heat treatment, and finally the surface was polished with 00000 grade emery cloth.

For constant stress amplitude tests the staircase statistical method for determining the fatigue limit was adopted using a stress interval of 0.5 kg per sq mm.

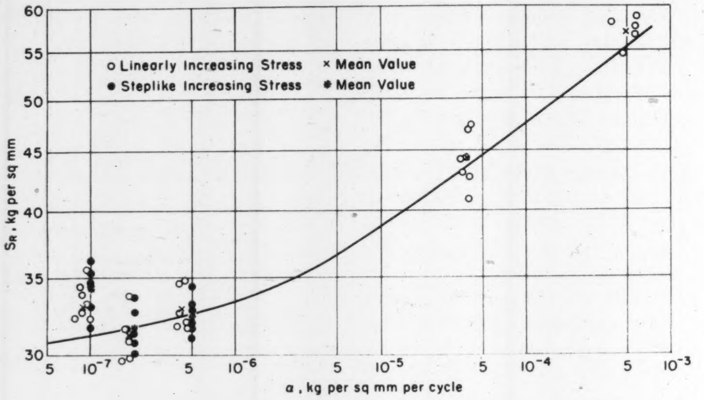


FIG. 4.—S_R *versus* α Diagram for S 10 Carbon Steel.

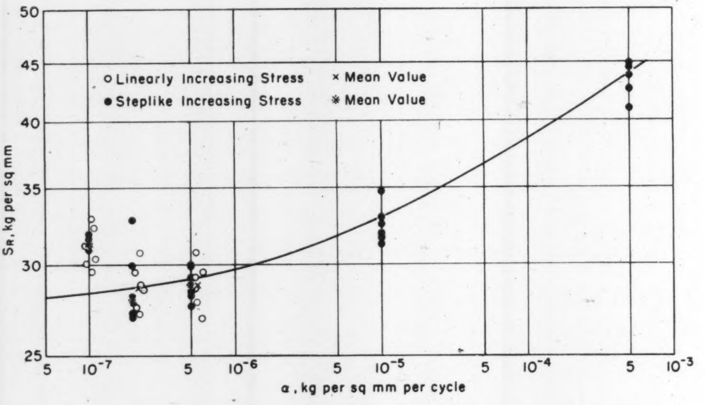


FIG. 5.—S_R *versus* α Diagram for S 40 Carbon Steel.

In the progressive loading fatigue test, the starting stress was selected at about 90 per cent of the endurance limit. However, when the loading rate was 10^{-7} kg per sq mm per cycle, the starting stress was selected at about 95 per cent of the endurance limit to reduce the time required for the test.

The *S-N* fatigue data procured under constant stress amplitude conditions are plotted in Fig. 2 for S10 carbon steel and in Fig. 3 for S40 carbon steel. From these diagrams, the value of S_e, N_c, and m were determined for each material. Figures 4 and 5 show the test results under progressive load increase in which full curves show the relations obtained from Eq 2 substituting the above characteristic values. Most of the mean points are considered to be on the curves except the groups of points lying in the range of $\alpha < 10^{-7}$ kg per sq mm per cycle, which deviate from the curves, indicating high rupture stress. This is considered to arise from the fact that the specimens had been strengthened by understressing. Therefore, about 2×10^{-7} kg per sq mm per cycle was obtained for both materials as a value of critical rate of stress increment.

FATIGUE TEST UNDER STEPWISE INCREASING ALTERNATING LOADS

Tests were carried out to determine whether there were any differences in the magnitude of stresses for failure between the case using linearly increasing load and one using stepwise increasing load.

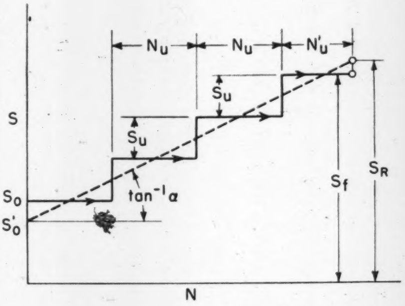


FIG. 6.—Stepwise Increased Load.

The mean rate of stress increment under stepwise loading was taken as the value shown in dashed line in Fig. 6, and the starting stress, S_o, was set as $S'_o + S_u/2$. The stress for failure under stepwise loading was converted into the one under linearly increased loading by the following equation:

$$S_R = S_f - \tfrac{1}{2}S_u + N'_u\alpha \ldots\ldots (3)$$

The results of these tests were also shown in Figs. 4 and 5.

In order to test whether there was a significant difference in the failure stress under linearly increasing load and the one under stepwise increasing load, some test points near to the lines of $\alpha = 2 \times 10^{-7}$ and 5×10^{-7} kg per sq mm per cycle were modified and moved to these lines. Modification was done as follows: Tangent lines were drawn to the calculated curve in Figs. 4 and 5 at $\alpha = 2 \times 10^{-7}$ and 5×10^{-7} kg per sq mm per cycle. The test points in the neighborhood were moved to these two values of α by moving the points parallel with these tangent lines. These modified values are listed in Table IV. Contingency 2 by 2 tables were obtained from this table for the test of significance and are shown in Table V. In this table, the terms "upper" and "lower" denote the number of points respectively located in the upper and lower region on either side of the median line. It is clear without resorting to test that there is no significant difference in the two lots of values in Table V (*a*), (*b*), and (*d*); the data in Table V (*c*) was found to exhibit no significant difference at the 5 per cent level of significance by testing with a binomial probability paper.

A SIMPLIFIED METHOD FOR PREDICTING THE WÖHLER FATIGUE LIMIT BY MAKING USE OF THE PROGRESSIVE LOADING METHOD

Figure 7 shows S_R/S_e *versus* α curves which have been replotted from all of the results of fatigue tests under progressively increasing loading that were gathered by the author from the literature. The data on the above mentioned materials and references are listed in Table II. In Fig. 7 the values of S_R/S_e are shown to decrease with a decrease of α except for some of the steels for which S_R/S_e increases for a decrease of α in the region of α less than 10^{-6} kg per sq mm per cycle. In the range $\alpha \geq 10^{-6}$ kg per sq mm per cycle, none of the materials was strengthened by understressing. The thick full curve and the thick dashed curve in the range between $\alpha = 10^{-6}$ and 10^{-5} kg per sq mm per cycle in this diagram show the mean values of the individual curves for steel and aluminum alloy, respectively. In

TABLE II.—MATERIAL DATA FOR FIG. 7.

Curves of Fig. 7	Material	Chemical Composition, per cent								Heat Treatment,[a] deg Fahr	Alternating Load[b]	References
		IRON and STEEL										
		C	Si	Mn	P	S	Ni	Cr				
1....	SAE 2340	0.40	0.28	0.74	0.019	0.020	3.48	...		1500 OQ, 900T	RB	Sinclair (4)
2....	SAE 1045	0.44	0.21	0.75	0.014	0.021	...	...		1500 N	RB	
3....	Ingot iron	0.012	...	0.017	0.005	0.025	...	...		1400 A, Aged	RB	
4....	Ingot iron	0.012	...	0.017	0.005	0.025	...	...		1400 WQ, Unaged	RB	
5....	SAE 4340	0.40	0.27	0.70	0.04	0.04	1.8	0.8	Mo 0.25	$H_v = 360$	RB	Ward, Schwartz & Schwartz (5)
6....	Welded SAE 4340	0.40	0.27	0.70	0.04	0.04	1.8	0.8	Mo 0.25	$H_v = 360$	RB	
7....	SAE 2340	0.40	0.28	0.74	0.019	0.020	3.48	...		1450 OQ, 1200T	RBC	Corten, Dimoff & Dolan (6)
8....	SAE 2340 notched	0.40	0.28	0.74	0.019	0.020	3.48	...		1450 OQ, 1200T	RBC	
9....	S40C V-notch	0.34	0.13	0.51	0.024	0.040	...	...		IH	RB	Enomoto (7)
10....	S40C	0.39	0.12	0.48	0.031	0.040	...	...		1560 N	RB	
11....	S10C	0.12	0.28	0.53	0.034	0.017	...	...		1400 WQ, Aged	RB	
12....	S40C	0.34	0.13	0.51	0.024	0.040	...	...		IH	RB	
13....	Spring steel	0.85	0.31	0.50	0.035	0.034	0.11	0.04	Cu 0.23	1500 OQ, 390T	RB	
14....	SAE 1020	0.2	...	0.45	0.45	0.055	...	...			RBC	Vitovec & Lazan (8)
15....	SAE 4340	0.38	0.30	0.74	0.015	0.021	0.73	0.75	Mo 0.24	1440 WQ, 1050T	RBC	
16....	35 CM 4	0.35	...	0.9	...	...	...	...			RB	Ferro & Rossetti (9)
17....	Ingot iron	0.012	...	0.017	0.005	0.025	...	...		1400 WQ, Not aged	RBC	Corten, Dimoff & Dolan (6)
18....	14 B 50	0.52	0.27	0.84	0.011	0.030	...	...	B 0.00005	1550 OQ, 550 T	RBC	
19....	Carbon steel	0.38	0.13	0.50	0.017	0.022	...	...			RB	Bastenaire, Cazaud & Weisz (10)
		TITANIUM										
20....	RC 55	C 0.45, N 0.067, H 0.0044, Ti Balance								1300 A	RBC	Vitovec & Lazan (8)

		ALUMINUM ALLOY			
21....	75S-T6	Zn 5.6, Mg 3.5, Cu 1.6, Cr 0.3	As received	RB	Sinclair (4)
22....	Duralumin	Cu 3.77, Mg 0.77, Mn 0.03, Fe 0.50, Si 0.39	930 WQ, Aged	RB	Enomoto (2)
23....	2024-T4	Cu 4.20, Mg 1.66, Mn 0.63, Fe 0.30, Si 0.14, Zn 0.07, Cr 0.02, Ti 0.02		RBC	Vitovec & Lazan (8)
24....	Duralumin	....	As received	RB	Ferro & Rossetti (9)
25....	75S-T6	Zn 5.6, Mg 3.5, Cu 1.6, Cr 0.3	As received	RBC	Corten, Dimoff & Dolan (6)
		BRASS			
26....	70-30 brass...	....	......	RB	Sinclair (4)
27....	70-30 brass...	Cu 67.21, Zn Balance	930 A	RB	Enomoto (2)

[a] OQ = oil-quenched; T = tempered; N = normalized; A = annealed; WQ = water-quenched.

[b] RB = rotary bending; RBC = rotary bending, cantilever type.

addition, the standard deviation of test data for iron and steel were calculated.[5] The 95 per cent confidence limits are indicated with short segments in this diagram. These calculated data are presented in Table III. From the above diagram, it is understood that the degree of scatter of the values of S_R/S_e for iron and steel is comparatively small in the region of $\alpha = 10^{-6}$ kg per sq mm per cycle.

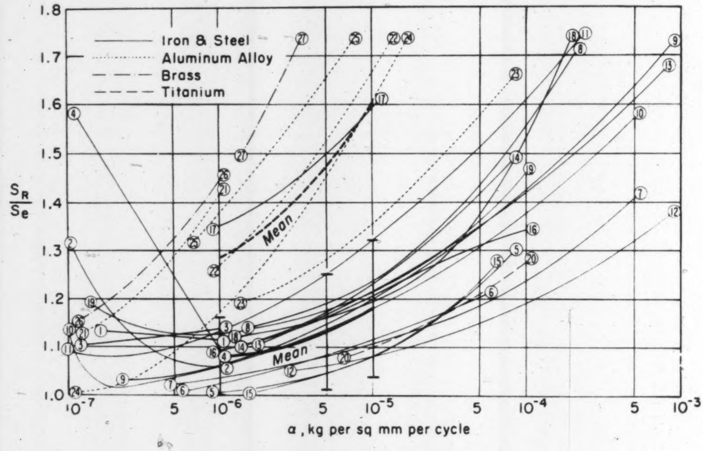


FIG. 7.—S_R/S_e *versus* α Diagram.
The "I's" Show 95 Per Cent Confidence Limit.

Thus, if the stress for failure, S_R, is determined experimentally for an iron or a steel specimen by using $\alpha = 10^{-6}$ kg per sq mm per cycle and is divided by 1.08 which is taken from Table III, a value close to S_e, with an error less than about ±8 per cent, may be obtained. Thus, the endurance limit may be predicted by testing a single specimen. If $\alpha = 5 \times 10^{-6}$ or 10^{-5} kg per sq mm per cycle is used for the test, the degree of confidence associated with the predicted values of S_e is lower as is seen from Fig. 7 and Table III, but on the other hand the test period can be reduced.

The degree of confidence in the various values for aluminum alloy in Table III is small because the test data are very few.

TABLE III.—CALCULATION OF DEGREE OF SCATTER.

α, kg per sq mm per cycle.....	10^{-6}	5×10^{-6}	10^{-5}
	IRON AND STEEL		
S_R/S_e (Mean value)....	1.081 +0.055 −0.076	1.131 +0.119 −0.086	1.180 +0.140 −0.099
$2\ \sigma^a$	0.081	0.120	0.142
	ALUMINUM ALLOY		
S_R/S_e (Mean value)....	1.281 +0.133 −0.108	1.477 +0.126 −0.195	1.602 +0.180 −0.247

[a] σ is standard deviation.

The starting stress should be somewhat smaller than the expected fatigue limit, which may be estimated from the ultimate strength of the material and experience; however, if an extended testing time is permitted, it is desirable to start the test from a smaller stress. If from the results of the tests the predicted fatigue limit is smaller than the starting stress, the starting stress should be regarded as having a large probability of exceeding the Wöhler fatigue limit.

---

[5] It is considered inappropriate to apply the statistical calculation in this instance, however the author used it on purpose to see the tendency of scatter.

It is desirable that the unit load in the stepwise increasing load tests be as small as possible, but if the unit load must be changed during the test for certain reasons, it should be carefully arranged so that the mean rate of stress increase is not changed. The stress for rupture must be corrected by Eq 3.

TABLE IV.—COMPARISON OF FAILURE STRESS (KG PER SQ MM).

$\alpha = 2 \times 10^{-7}$ kg per sq mm per cycle		$\alpha = 5 \times 10^{-7}$ kg per sq mm per cycle	
S10 C—Steel	S40 C—Steel	S10 C—Steel	S40C—Steel
UNDER LINEARLY INCREASED LOAD			
33.8	30.7	34.9	30.6
31.7	29.5	34.8	29.3
31.7	28.7	32.8	29.2
30.8	28.4	32.1	28.6
29.9	27.4	32.0	27.7
29.7	27.1	31.6	26.7
UNDER STEP-LIKE INCREASED LOAD			
33.6	32.8	34.3	29.9
32.7	29.9	33.8	29.6
31.4	28.1	32.8	29.3
31.2	27.7	32.0	28.4
30.7	27.2	31.6	28.3
30.1	26.9	29.9	27.6
Table V (*a*)	Table V (*b*)	Table V (*c*)	Table V (*d*)

SUMMARY AND CONCLUSIONS

A simplified method was appraised for predicting the Wöhler fatigue limit by correcting the stress for failure in a progressive loading fatigue test using only one loading rate. The ratio of stress increase which can be used, the ratio between the stress for rupture and the Wöhler fatigue strength, and the degree of confidence of the predicted fatigue strength were determined and discussed. It is desirable that the degree of confidence associated with the predicted fatigue strength be increased by additional experimental verification.

It was confirmed by experiments that when stepwise increasing loading was used for fatigue tests instead of linearly increasing loading, there was no substantial difference between the values of the failure stress. Also when this stepwise increasing loading method is used, the test can be performed with a conventional fatigue testing machine without adding a particular device to effect continuously increasing loading.

TABLE V.—CONTINGENCY 2 × 2 TABLE.

	S_R	S_R Step	Total	S_R	S_R Step	Total
(*a*) S10 C—Steel, $\alpha = 2 \times 10^{-7}$ kg per sq mm per cycle				(*b*) S10 C—Steel, $\alpha = 5 \times 10^{-7}$ kg per sq mm per cycle		
Upper......	3	3	6	3	3	6
Lower......	3	3	6	3	3	6
Total......	6	6	12	6	6	12
(*c*) S40 C—Steel, $\alpha = 2 \times 10^{-7}$ kg per sq mm per cycle				(*d*) S40 C—Steel, $\alpha = 5 \times 10^{-7}$ kg per sq mm per cycle		
Upper......	4	2	6	3	3	6
Lower......	2	4	6	3	3	6
Total......	6	6	12	6	6	12

Furthermore, in using this method, the fatigue limit of large specimens or of construction members may be determined because the prediction of the fatigue limit is possible using very few specimens.

Acknowledgments:

The author wishes to express his deep gratefulness to T. Hara for his general support of the investigation and to S. Murayama for his assistance and cooperation.

REFERENCES

(1) E. M. Prot, "L'Essai de Fatigue sous Charge Progressive," *Comptes Rendus,* Vol. 225, p. 669 (1947).

(2) N. Enomoto, "On Fatigue Tests under Progressive Stress," *Proceedings,* Am. Soc. Testing Mats., Vol. 55, p. 903 (1955).

(3) N. Enomoto, "An Application of the Idea of Cumulative Damage to the Results of Fatigue Tests under Progressive Load," *Proceedings,* The 1st Japan National Congress for Testing Materials, p. 45 (1957).

(4) G. M. Sinclair, "An Investigation of the Coaxing Effect in Fatigue of Metals," *Proceedings,* Am. Soc. Testing Mats., Vol. 52, p. 743 (1952).

(5) E. J. Ward, R. T. Schwartz, and D. C. Schwartz, "An Investigation of the Prot Accelerated Fatigue Test" *Proceedings,* Am. Soc. Testing Mats., Vol. 53, p. 885 (1953).

(6) H. T. Corten, T. Dimoff, and T. J. Dolan, "An Appraisal of the Prot Method," *Proceedings,* Am. Soc. Testing Mats., Vol. 54, p. 875 (1954).

(7) N. Enomoto, "Study on Fatigue Life of Metals by Means of Progressive Stress Fatigue Testing, *Journal of Railway Engineering Research* (Japan), Vol. 12, p. 363 (1955).

(8) F. H. Vitovec and B. J. Lazan, "Strength, Damping and Elasticity of Materials Under Increasing Reversed Stress with Reference to Accelerated Fatigue Testing," *Proceedings,* Am. Soc. Testing Mats., Vol. 56, p. 844 (1956).

(9) A. Ferro and U. Rossetti, "Contribution à l'Étude de la Fatigue des Matériaux avec Essais à Charge Progressive," *Colloquium on Fatigue,* Springer Verlag, Berlin p. 24 (1956).

(10) F. Bastenaire, R. Cazaud, and M. Weisz, "Essais de Fatigue Statistiques Suivant la Methode de Charge Progressive," *Colloquium on Fatigue,* Springer Verlag, Berlin, p. 14 (1956).

DISCUSSION

Mr. Ronald F. Brodrick.[1]—The author has made another of his several fine contributions to the use of accelerated methods of fatigue testing.

We have employed the Prot method in the past and found it useful in cases where the number of specimens available is sufficient to establish the slope of the Prot relation. Once the slope is established, testing can continue at a single rate of stress increase, thus reducing testing time. The method is especially valuable for comparative work, since relative strengths can be established with a reasonable degree of confidence, even though absolute values may not be determined with precision.

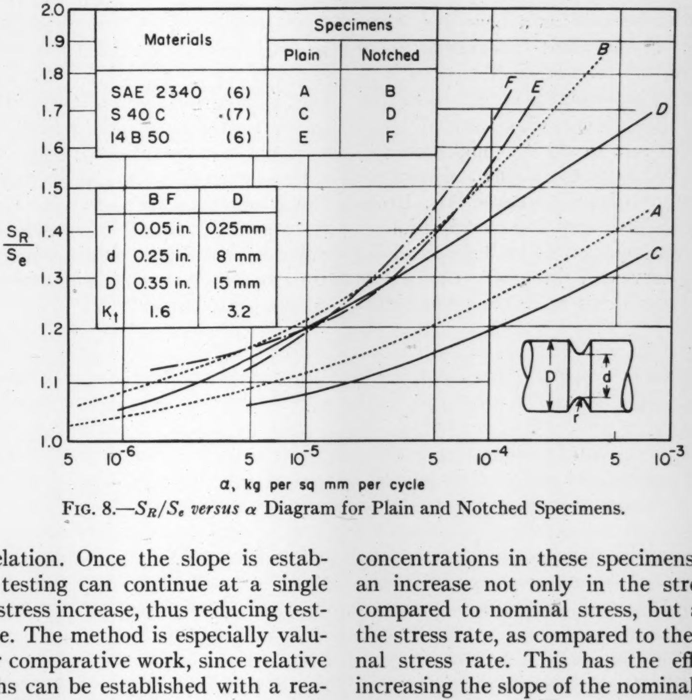


Fig. 8.—S_R/S_e *versus* α Diagram for Plain and Notched Specimens.

The Prot method or modifications presented by the author would appear very attractive in the testing of complex, expensive components, assuming the Prot slope is known for the parent material. One must note however that any stress concentrations in these specimens cause an increase not only in the stress, as compared to nominal stress, but also in the stress rate, as compared to the nominal stress rate. This has the effect of increasing the slope of the nominal stress *versus* nominal $\sqrt{\alpha}$ curve over that of the parent material. This increase is a function of the strength reduction factor.

---

[1] Senior Project Engineer, Lessells and Associates, Inc., West Bridgewater, Mass.

Failure to take this into account when single values of α are to be used in testing could lead to sizeable underestimates of fatigue limit. Thus, application of either stepwise or continuous load increases to specimens with stress concentrations would seem to require prior knowledge of strength reduction factors, if the characteristics of the parent material are to be used as a base line. Conversely, if sufficient specimens were available, the methods could potentially be used to determine strength reduction factors. Additional experimental work along this line would be enlightening.

TABLE VI.—K_t AND K_f FOR NOTCHED SPECIMENS.

Material	K_t	K_f
SAE 2340	1.6	2.0
S40C	3.2	2.5
14B50	1.6	1.6

MR. N. ENOMOTO (*author's closure*).—The author wishes to thank Mr. Brodrick for his comment and contribution.

The accompanying Fig. 8 shows S_R/S_e *versus* α curves for three parent materials and for specimens with stress concentrations. Since the ordinate is shown as S_R/S_e, this value will not vary in regions of stress concentration as compared with the nominal value. The rate of stress increase, α, however, increases in the stress concentration region as compared with the nominal stress rate. Consequently, the S_R/S_e *versus* α curve calculated for the stress concentration region is displaced to the right of that for the nominal stress and is to approach that for the parent material. However, the effect of strength reduction due to stress concentration is weakened as the stress rate becomes larger, and the curve is considered to move upward from that of the parent material. Accordingly, if the fatigue strength, S_e, for notched specimens is obtained making use of the S_R/S_e *versus* α curve for the parent material and the nominal stress rate, it would lead to an overestimate of fatigue limit.

Stress concentration factors K_t and fatigue strength reduction factors K_f for these materials are shown in Table VI. At present, it would be considered difficult to derive the strength reduction factor from S_R/S_e *versus* α curves or from S_R *versus* α^n curves.

EFFECT OF INTERNAL HEATING ON THE FATIGUE LIFE OF TITANIUM*

By J. P. Romualdi[1] and E. D'Appolonia[2]

Synopsis

This paper presents results of tests to study the effect of internal heating on the fatigue properties of titanium. A continuous time record of temperature and deflection was obtained during fatigue tests of specimens under isothermal and nonisothermal conditions. The tests were conducted at different stress levels and at speeds ranging from 1000 to 10,000 rpm.

The bending moment-deflection relationship for a specimen, before the start of the fatigue test, is that which would be predicted on the basis of properties determined from a standard tension test. After the first few cycles of repeated stress, however, for specimens stressed to a limited extent above the elastic limit, this deflection decreases to that which would be predicted from a linear elastic theory. If subsequent overheating due to rapid cyclic stressing is prevented, either by a coolant (isothermal test) or by slow testing speeds, this pseudoelastic deflection is maintained for almost the entire fatigue life of the specimen. Under nonisothermal conditions and rapid testing speeds, the deflection increases again and the temperature of the specimen rises. This temperature rise causes a thermal softening and the specimen has a ragged discolored fracture surface. If the stress is low enough there is no appreciable temperature rise and the specimen will either fail due to a spreading fatigue crack or run indefinitely.

Studies of fatigue on the phenomenological level are usually concerned with the number of cycles of stress a material can withstand without failure by fracture. However, the behavior of a material while it is being subjected to cyclic stressing is also of great importance. Besides failure by fracture, failure can be defined by a maximum permissible distortion. Also, it is of interest to know when, during the finite fatigue life, the actual destruction of a material begins. Crum and D'Appolonia (**1**),[3] for example, used knowledge of the deflection variation during cyclic stressing to determine the damage line for titanium.

Earlier experiments on the behavior of materials during cyclic stressing were performed by Bairstow (**2**). He observed that one cycle of complete stress reversal to ±31,600 psi on axle steel showed no observable hysteresis loop. However, after 18,750 cycles of stress an hysteresis loop had formed and the width of this loop was 11 per cent of the original elastic extension. He also observed that when the stress was not completely re-

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Assistant Professor, Department of Civil Engineering, Carnegie Institute of Technology, Pittsburgh, Pa.

[2] E. D'Appolonia Associates, Consulting Engineers, Pittsburgh, Pa.; Lecturer, Carnegie Institute of Technology, Pittsburgh, Pa.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

versed a permanent extension occurred in addition to the hysteresis loop. Experiments by Gough (3) indicate that specimens showing an hysteresis loop in the first cycle of reversal might, after several thousand cycles of stress below the endurance limit, show no appreciable hysteresis loop. Experiments have shown that this approach to a purely elastic condition is slow and that the energy dissipated per cycle might be sufficient to cause an appreciable temperature rise during the test. Gensamer (4) interprets this phenomenon as indicating that fatigue is a "... race between hardening and damage...."

These experiments and others indicate that "observables" such as temperature and deflection of fatigue specimens offer valuable insight into the behavior of metals when subjected to cycles of repeated stress. This paper reports on a study of these "observables" in titanium fatigue specimens tested at different speeds and at different stress levels.

MATERIAL USED

The material used was $\frac{5}{8}$-in. diameter RC-55 titanium rod. This material was manufactured by Republic Steel Corp. and is of the type referred to as "commercially pure." It has a nominal composition of 0.156 per cent carbon, 0.040 per cent nitrogen, traces of impurities and the remainder titanium.

TESTING APPARATUS

A program was carried out to obtain quantitative results of the effect of internal heating on the fatigue properties of titanium. An apparatus was built to investigate the relationship between temperature and deflection during fatigue tests conducted at different speeds and different stresses.

FIG. 1.—Photograph of Testing Apparatus.

The apparatus is shown in Fig. 1. Figure 2 is a schematic diagram of the temperature and deflection recording units. A chrome-alumel thermocouple held in contact to the smallest section of the specimen by a fine wire measured the temperature. Contact between the ther-

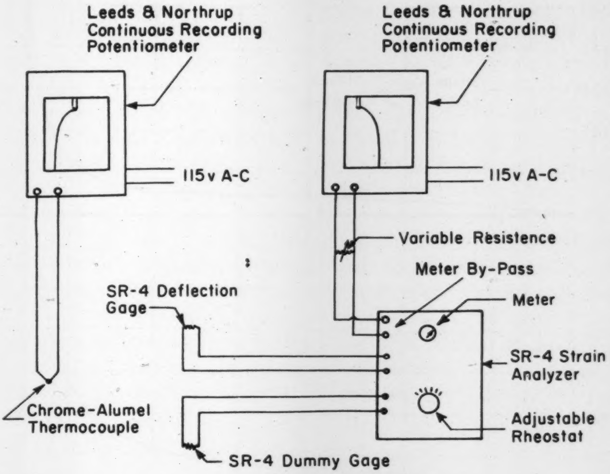

Leeds & Northrup Continuous Recording Potentiometer
Leeds & Northrup Continuous Recording Potentiometer
115v A-C
115v A-C
Variable Resistence
Meter By-Pass
SR-4 Deflection Gage
Meter
SR-4 Strain Analyzer
Chrome-Alumel Thermocouple
Adjustable Rheostat
SR-4 Dummy Gage

FIG. 2.—Schematic Diagram Showing Temperature and Deflection Recording Apparatus.

mocouple and the leads to the temperature recorder was accomplished by means of two rotating contacts mounted on the specimen. A layer of bakelite insulated the contacts from the specimen.

The deflection recorder consisted of an electric-resistance strain gage mounted on a reed of thin spring steel. One end of the reed was fastened to a wooden block clamped to the base of the R. R. Moore fatigue testing machine. The other end of the reed was in contact with the bottom of the loading harness.

The strain gage was connected to an SR-4 strain analyzer as shown in Fig. 2. The current by-passed the meter on the analyzer and was measured by a continuous recording potentiometer. A variable resistance was mounted in parallel with the recorder to regulate the current passing through it. For a given maximum deflection of the specimen, the total sweep of the recorder was adjusted to give maximum sensitivity of operation. The deflection gage was calibrated with a micrometer accurate to 0.001 in.

Standard R. R. Moore fatigue specimens were used with a slightly longer gage length to accommodate the temperature recording device.

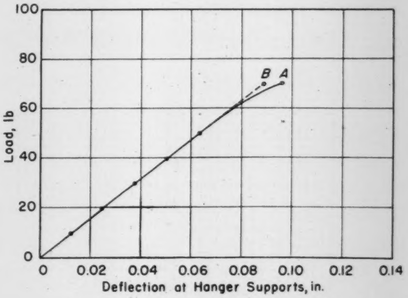


FIG. 3.—Load-Deflection Tests on Titanium.

RESULTS OF TESTS

Figure 3 shows the results of a load-deflection test on a titanium specimen mounted in the fatigue machine. The solid line represents the load-deflection test before the onset of cyclic stressing. Above loads of 50 lb the outer fibers of the specimen were stressed into the plastic range as shown by the nonlinear

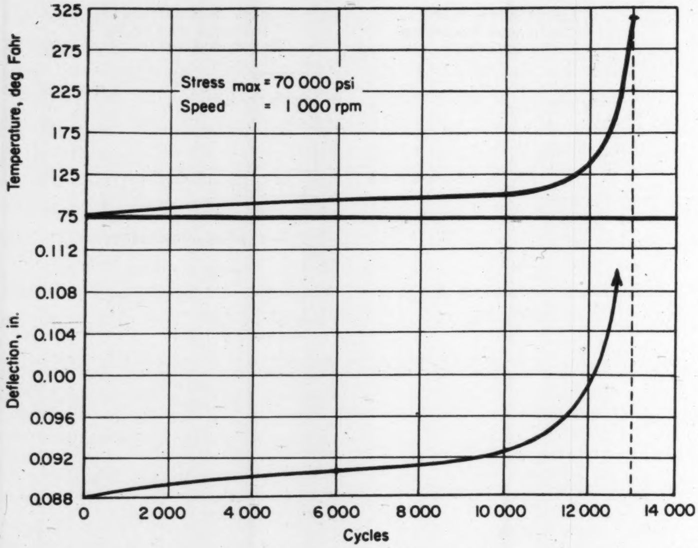


FIG. 4.—Plot of Change in Deflection and Temperature Against Number of Cycles.

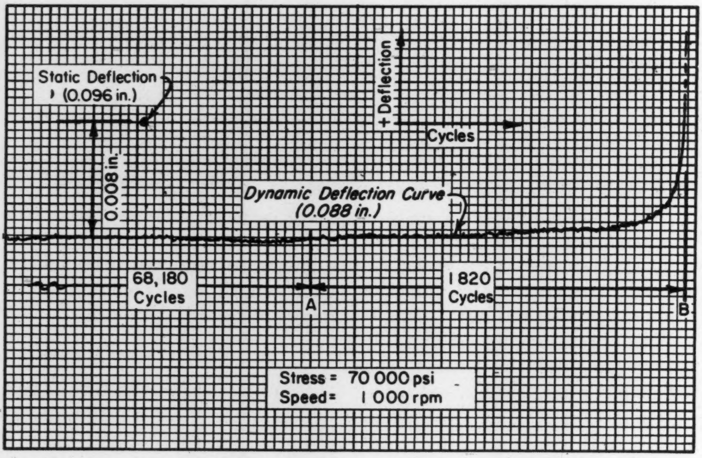


FIG. 5.—Deflection Curve Near Fracture for Isothermal Fatigue Test of Titanium.

portion of the curve. A load of 71.5 lb produced the deflection 0.096 in. indicated by point *A* on the diagram. This load corresponds to a nominal maximum stress of 70,000 psi. After the fatigue test was started, the deflection recorder indicated a decrease in deflection, after the first few cycles, to 0.088 in. When this smaller deflection is plotted on the load-deflection diagram (point *B*) it lies on a straight line with the linear portion of the curve. Thus, within the first few cycles of stress, the yield point is raised and it seems reasonable to conclude that the specimen behaves elastically. Results similar to this obtained from a different testing apparatus have been reported by the authors in an earlier paper (5).

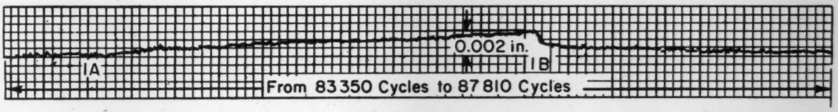


Fig. 6.—Portion of Deflection Curve for Isothermal Fatigue Test. Stress, 70,000 psi; speed, 1000 rpm.

Figure 4 shows that this apparent linear elastic condition is not maintained throughout the whole of the fatigue test. The figure is a plot of temperature and deflection as a function of number of cycles of stress for the specimen described above tested at 1000 rpm. The decrease in deflection from 0.096 in. to 0.088 in. is not shown inasmuch as it occurred in only three or four cycles. The test is shown to start at the apparent elastic deflection of 0.088 in. Subsequent to the decrease to 0.088 in., Fig. 4 shows the deflection began gradually to increase again. This gradual increase in deflection was accompanied by an increase in temperature. Only after 11,500 cycles of stress did the deflection reach the initial elastic-plastic deflection of 0.096 in. indicated by point *A* in Fig. 3.

The question now arises as to what effect the temperature rise had upon the deflection. In other words, had the specimen been kept at a constant temperature would the deflections have been similar to those recorded? Tests, to evaluate this effect, were conducted under isothermal conditions by running water as a coolant through a water jacket surrounding the specimen.

As in the nonisothermal tests, the chart recorder for the controlled temperature tests indicated a sudden decrease in deflection at the start of the test. Figure 5 is a part of the chart record showing a portion of the deflection curve of a specimen stressed to 70,000 psi and tested isothermally at a speed of 1000 rpm. As in the test described above, the initial decrease in deflection was 0.088 in. After attaining this minimum deflection the specimen underwent 68,180 cycles of stress without any further change. At point *A* in Fig. 5 the specimen began to deflect and fracture followed after 1820 additional cycles of stress. Note that under controlled isothermal tests the initial elastic-plastic deflection was not obtained until the last few cycles before fracture.

The fracture surfaces of the fatigue specimens fractured under nonisothermal conditions at high testing speeds were jagged and discolored. The surfaces of specimens fractured under isothermal conditions were smooth and exhibited a typical fatigue fracture. The deflections during isothermal tests were constant, and only near the end of the finite fatigue life were the deflections pronounced. Under nonisothermal conditions the deflections increase in the early stages of

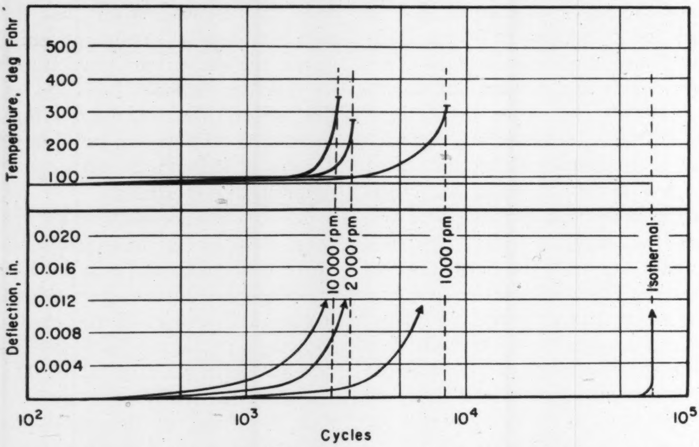


FIG. 7.—Temperature and Deflection Tests at a Stress of 70,000 psi under Different Test Conditions.

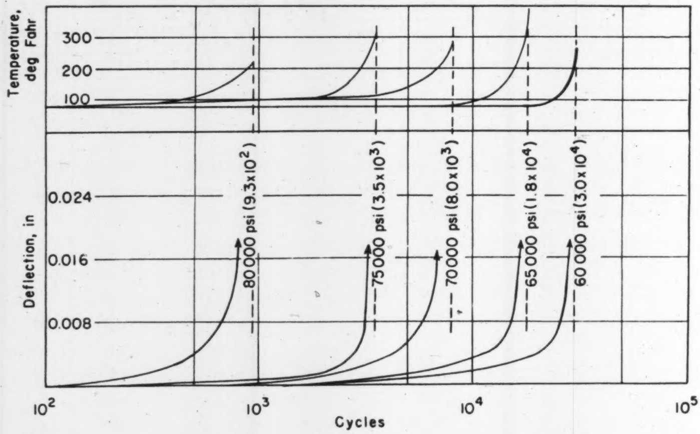


FIG. 8.—Temperature and Deflection Tests at 1000 rpm at Different Stress Levels (Nonisothermal).

testing and the finite life is appreciably shortened. This leads one to believe that the deflections observed during nonisothermal tests are principally due to a thermal softening effect.

This is supported by the data presented in Fig. 6 which shows a portion of the chart from the deflection recorder. The test is isothermal. At point *A* the circulating cooling water was shut off and the deflection of the specimen began to increase. At point *B* the water was recirculated and the deflection quickly returned to the value at point *A*. No permanent deflection could be detected.

The results of temperature-deflection tests conducted at different speeds and different stress levels are summarized in

TABLE I.—ISOTHERMAL TESTS AT 70 000 psi.

Specimen	Speed, rpm	Cycles to Failure
2 E-2	10 000	65 000
2 F-2	10 000	74 000
2 E-3	2 000	49 000
2 E-7	2 000	78 000
2 E-4	1 000	70 000

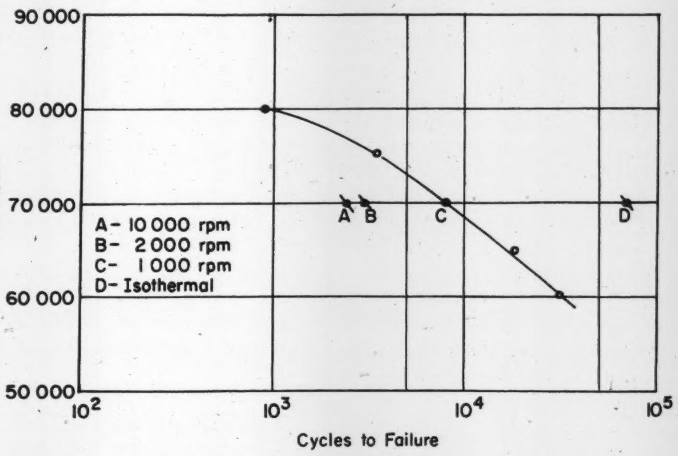


FIG. 9.—*S-N* Diagram for Varying Stress - Constant Speed Tests and Constant Stress - Varying Speed Tests.

Figs. 7 and 8. Each curve is the average of three or more tests. Figure 7 shows the results for specimens tested at complete stress reversal at a maximum stress of 70,000 psi without coolant and at different speeds—10,000, 2000, and 1000 rpm. Isothermal tests were conducted only at 1000 rpm. Figure 8 shows results for specimens tested at 1000 rpm at stress levels ranging from 60,000 to 80,000 psi.

Isothermal tests conducted at different testing speeds from 1000 to 10,000 rpm showed no appreciable difference in the finite fatigue life. Results are within the usual scatter as shown in Table I.

DISCUSSION OF RESULTS

Static load-deflection tests conducted on specimens in the fatigue machine before the test was started disclosed plastic yielding at high loads (corresponding to stresses above the endurance limit). However, when the fatigue machine was turned on, the deflection of the specimens immediately decreased to a value consistent with a linear stress-strain relationship. Moreover, this elastic deflection was maintained for about 90 per cent of the life of the specimens if the tests were isothermal.

In the case of nonisothermal tests, however, the material behaved quite differently. After the first few cycles of stress, the specimens began to deflect again. A rise in temperature was observed with this increase in deflection. If the temperature rise was not checked, as in the isothermal tests, a thermal softening of the material occurred.

Associated with this thermal softening is an increase in the area of the hysteresis loop. The amount of heat generated per cycle per unit volume is the area under this hysteresis loop. Therefore, as the area of the loop increases the amount of heat generated per unit of time, for a given testing speed, also increases. In order to dissipate this excess heat the temperature of the specimen increases again and there is a further decrease in mechanical properties. This destructive action continues until the specimen fails. Fractures of this kind are characterized by a rough, jagged, discolored section.

By cooling the specimen (checking the temperature rise) at any stage during a test for a certain range of stresses, the deflections could be decreased to the linear elastic deflection that existed after the first few cycles of stress. This indicates that the deflections occurring during the nonisothermal tests are primarily due to thermal softening and not crack propagation. This is supported by the fact that specimens tested under isothermal conditions maintain their original elastic deflection for about 90 per cent of the finite fatigue life.

The lower bound of the endurance limit, for increasingly higher testing speeds, is not well defined. It may be argued that the lowest stress level for the endurance limit is the stress at which the material behaves perfectly elastically (no hysteresis loop). But equally well it may be argued that such a condition is never realized and slight hysteresis loop will always occur (this is sometimes called elastic hysteresis). Therefore, no matter how small the hysteresis loop there is some high testing speed where the rate of heat generation per unit time is high enough to cause a temperature rise that will bring about thermal softening.

The effects of speed of testing on the finite fatigue life of specimens tested under different conditions is summarized in Fig. 9. This figure presents the test results shown in Figs. 7 and 8 in the more familiar form of an S-N diagram. Curve C shows the results of specimens tested at different stresses, but at the same testing speed of 1000 rpm. Points A, B, and D indicate how far this curve can be shifted if the specimens are tested at 10,000 rpm, 2000 rpm, or isothermally. These differences due to internal heating are much larger than the differences due to strain-rate effects which would be observed if the temperature had been controlled (isothermal tests).

Acknowledgments:

This work was done in connection with a research program conducted at Carnegie Institute of Technology for the Office Chief of Ordnance, Research and Development Branch under contract No. DA-36-061-ORD-259 on a study of the fatigue characteristics of titanium. The work was under the technical supervision of the Applied Mechanics Branch of Watertown Arsenal, Watertown, Mass.

REFERENCES

(1) R. G. Crum and E. D'Appolonia, "Behavior of Ti-75A Titanium Alloy Under Repeated Load," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, pp. 969–978 (1955).

(2) L. Bairstow, "The Elastic Limits of Iron and Steel Under Cyclic Variations of Stress," *Philosophical Transactions*, Royal Society (London), Vol. 210a, p. 35 (1910).

(3) H. J. Gough, "The Fatigue of Metals," Ernest Benn, Ltd., London (1926).

(4) W. M. Murrey, "Fatigue and Fracture of Metals," John Wiley & Sons, Inc., New York, N. Y. (1950).

(5) J. P. Romualdi and E. D'Appolonia, "Effect of Range of Stress and Prestrain on the Fatigue Properties of Titanium," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, pp. 798–821 (1954).

DISCUSSION

Mr. C. E. Feltner[1] (*presented in written form*).—The authors are to be congratulated on their work. The effect of internal heating on the fatigue life of this material is clearly observable from the type of experimental test that has been employed.

There are three aspects of this paper that I would like to briefly discuss; the first is the statement by the authors that the area under the mechanical hysteresis loop is equal to the amount of heat generated per cycle per unit volume of material. To my knowledge this statement may be somewhat incorrect since some investigators have shown that at comparatively high stresses about 90 per cent of the energy dissipated per cycle goes off in the form of heat (**1**),[2] the remainder contributing to a change in internal energy.

The second aspect concerns the statement, "It may be argued that the lowest stress level for the endurance limit is the stress at which the material behaves perfectly elastically (no hysteresis loop)." If a material were perfectly elastic, then there would be no deterioration of the elastic energy and no hysteresis loop. Internal friction tests at stresses as low as 10 psi (**2**) show that there is a dissipation of energy and thus a hysteresis loop exists. The fact that most of our strain measuring equipment cannot detect the anelastic and microplastic strains which exist has led to the common fallacy that there is some stress at which a material will behave perfectly elastically.

The third aspect deals with the authors' conclusion that thermal softening is the primary reason for the noted increase in deflection and decrease in fatigue life. I certainly agree with this conclusion but do feel that the conclusion might be further clarified. It is difficult to separate the difference between the effect of thermal softening and the effect of strain rate, for the thermal softening would not be present if low strain rates were used. An isothermal test could be run by using a strain rate which approaches zero. However, the time required to complete such a test would approach infinity which is not feasible. This reasoning does serve to show that thermal softening is inherently related to strain rate.

The authors' means of obtaining internal heating of the specimen is by increasing the frequency. Data obtained by Lazan and Wu (**3**) on mild steel show the effect of frequency on the damping energy, ΔW, dissipated per cycle per unit volume of material at a constant stress. It was found that an increase in frequency causes a decrease in ΔW which is generally true for most materials. However, the heat generated per cycle is being collected at a greater rate. Since a finite length of time is required to transfer a unit of heat, an increase in temperature may occur when the frequency is increased if, A, the rate of heat generation exceeds the rate of heat transfer and condition A will depend on whether, B, the rate of heat generation is great enough to overcome the decrease in ΔW due to the increase in frequency. The authors' results show that both of these conditions are present since an increase in temperature was observed in their tests.

---

[1] Graduate Fellow, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this discussion.

The temperature rise will then cause an increase in ΔW (proportional to the heat dissipated) as shown by the results of Schabtach and Fehr (4). The increase of ΔW means an increase in temperature which further increases ΔW, etc. The runaway process becomes more and more divergent until fracture finally occurs. If the temperature is measured throughout the test, a record of temperature *versus* number of cycles would be obtained similar to Fig. 4 of the authors' work.

For the high stresses used in their work, plastic deformation is probably the dominant strain hysteresis mechanism causing internal heating. Anelastic strain hysteresis mechanisms would add only a small amount to the total internal heating effect.

An interesting observation of the results of this paper shows that the greater the energy dissipated per cycle the shorter is the fatigue life. This suggests that the total energy collected might be a criterion for the fatigue life.

MESSRS. J. P. ROMUALDI AND E. D'APPOLONIA (*authors*).—The authors' statement that the area under the hysteresis loop is equal to the amount of heat generated per cycle per unit volume should, according to Mr. Feltner's comments, be changed to read—"almost all the area." This would not change the conclusions or basic concepts since, according to Mr. Feltner, the heat dissipated is 90 per cent of the area. With respect to the second observation that there is actually a hysteresis loop as low as 10 psi, we are in complete agreement. The obvious conclusion, therefore, is that one could always test at a sufficiently rapid speed to cause heating and subsequent failure. In this case the endurance limit would be at zero stress level. Practically speaking, however, such speeds would be impractical and one would observe an apparent endurance limit.

In general, Mr. Feltner's comments are very welcome and follow closely the authors' thinking. We are pleased to note that the results have met with such good reception.

MR. HERBERT T. CORTEN.[3]—From the fatigue lives observed, these experiments fall into the category frequently referred to as low-cycle fatigue. In this range, excellent correlation has been obtained between plastic strain range per cycle and fatigue life. While it is true that the differences in life found at one stress level were caused by running the experiments at different speeds, it appears from examination of Figs. 7 and 8 that the effect of speed of testing may be only incidental to other more fundamental factors, namely, the plastic strain range and the temperature of the specimens. While all of these factors are intimately interrelated, it would appear desirable to obtain data from which one may choose the variables that are known to correlate with much other data. Have the authors, in contemplating further work of this nature, considered making experiments in which the plastic strain range as well as the nominal computed stress are measured in order to facilitate correlations with other data?

MESSRS. ROMUALDI AND D'APPOLONIA.—Studies of this nature would be a very appropriate extension of the work we have done. Our studies have merely considered the extent of the phenomenon for one material under a limited variety of test conditions. The next step would be to attempt correlations that could lead to more general conclusions and form a basis for predicting behavior.

---

[3] Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

MR. J. M. HOLT.[4]—In our experience at the Applied Research Laboratory with the heat generated by internal friction during fatigue testing, we have found that with most of the steels being tested in a rotating-beam machine at 10,000 rpm or in a Rayflex machine at 17,000 or 18,000 cpm, it is necessary to blow compressed air on the specimens to remove the heat caused by internal friction. No provision is made to precool the air. With respect to the adequacy of this method of cooling, we observed, by means of a thermocouple attached to a 1-in. in diameter round bar of 4340 steel 29 in. long being tested on a Rayflex machine, that the bar could be cooled from 150 to about 90 F in a few minutes.

MR. BENJAMIN J. LAZAN.[5]—I should like to comment on the damping properties of titanium alloys and the associated temperature increase in a specimen under cycle stress. We have studied the damping and fatigue properties of two titanium alloys, RC-55 annealed and RC-130B. The damping of RC-55 increases rather abruptly as the function of stress at stresses beyond 50 per cent of the fatigue limit. In this region, rather high temperature increases can be expected even though there is no apparent fatigue damage taking place. By contrast, the damping of RC-130B does not increase abruptly until stresses above the fatigue limit are reached. It is therefore rather difficult to generalize whether the abrupt increase in temperature is associated with fatigue damage, fatigue cracking, or plastic working at the crack front, or whether the hysteresis mechanism is a nondamaging one.

It is possible to calculate the temperature increase in a fatigue specimen if the shape, stress distribution, damping properties and heat transfer coefficients are given. Such calculations show that high temperatures can be produced in a fatigue specimen stressed in a safe range.

MESSRS. ROMUALDI AND D'APPOLONIA.—If damping increases abruptly at stresses beyond 50 per cent of the fatigue limit, then one could select a speed of testing that is high enough, say at a stress of 75 per cent of the endurance limit, to cause a relatively high specimen temperature. It is inconceivable that the specimen could not be made to fail in a finite length of time at this stress, by selecting a sufficiently high testing speed. Thus, the fatigue limit referred to is a function of the testing speed at which it was determined.

The observation that the temperature increase could be calculated from known damping, testing speed, and stress distribution is worthy of further consideration. Such studies, coupled with studies suggested above by Mr. Corten, could go far toward predicting finite fatigue life at high speed—or amount of coolant necessary to prevent failure.

---

[4] Mechanical Testing Group, U. S. Steel Corp., Applied Research Laboratory, Monroeville, Pa.

[5] Professor of Materials Engineering, University of Minnesota, Minneapolis, Minn.

REFERENCES

(1) A. L. Titchener and M. B. Bever, "The Stored Energy of Cold Work," *Progress in Metal Physics*, Chalmers and King, Vol. 7, Pergamon Press, pp. 247–338 (1958).

(2) C. Zener, "Elasticity and Anelasticity of Metals," University of Chicago Press, Chicago, Ill. (1948).

(3) B. J. Lazan and T. Wu, "Damping, Fatigue and Dynamic Stress-Strain Properties of Mild Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 647 (1951).

(4) D. Schabtach and R. O. Fehr, "Measurement of the Damping of Engineering Materials During Flexural Vibrations at Elevated Temperatures," *Journal of Applied Mechanics*, Vol. 11, No. 2, p. A-86 (1944).

CRACK PROPAGATION IN COLD-ROLLED ALUMINUM SHEET*

BY H. A. LIPSITT,[1] F. W. FORBES,[1] AND R. B. BAIRD[1]

SYNOPSIS

Low-cycle, high-stress fatigue tests in fluctuating tension were performed on transverse and longitudinal specimens of sheet 1100-H18 aluminum alloy. The kinetics of growth of a fatigue crack initiated at a mild semicircular notch were determined at several stresses from stop-motion photographic records.

The data indicate that the growth of a fatigue crack is not linear (on a semilogarithmic plot) in these tests, or even continuous. Crack growth is seen to consist of short bursts of growth followed by periods of no measurable growth. In the initial stages, the growth bursts are proportional to the stress and the rest periods inversely proportional.

It is paradoxical to note that while the greater portion of fatigue life is acknowledged to consist of the propagation of a crack, this portion of fatigue life is one of the least understood facets of this ubiquitous phenomenon. Recent review articles (**1**,**2**,**3**)[2] have pointed out the need for a better understanding of crack initiation and propagation. These same articles, however, have indicated that the approaches used to date have not yielded information of sufficient exactness to allow more than a semiempirical analysis to be obtained from them. In fact, the most difficult portion of such a study is to find a method of obtaining data that are sufficiently exact to show a complete picture of the actual propagation of the crack (see reference (**1**), Appendix A).

Although the "oyster shell" markings visible on the fracture surfaces of a fatigue specimen have long been taken as being indicative that such crack growth is not a continuous process, only scant other information points to this fact. Since such information is of paramount importance to any detailed analysis of the processes occurring in fatigue, an attempt was made to find a more precise method of following the propagation of a fatigue crack. The technique chosen was basically one of stop-motion photography with the instrumentation so arranged that a photograph of the propagating fatigue crack would be taken at the peak-stress instant of the cycle, and at any chosen frequency from one to ten cycles per photograph.

The resultant films were then analyzed to obtain the crack growth characteristics. Previous observations of several investigators have been corroborated and extended; other observations are shown to be open to question, mainly because their observational frequency was so low as to essentially smooth out the data.

Finally, a number of other observations made from the films and from examination of the specimens are reported. These observations and their impact are discussed, and several previously unreported facets of crack growth are presented.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Project Scientist, Metallurgy Research Branch, Engineer-in-Training and Task Scientist of the Mechanics Research Branch, respectively, Aeronautical Research Laboratory, Directorate of Laboratories, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

LITERATURE REVIEW

The bulk of the literature on crack initiation and propagation has been surveyed (1,2,3). The conclusions of these reviews may be summarized as: (*a*) cracks initiate later in life as the surface finish is improved, as the temperature is increased, and as the stress is decreased; (*b*) crack growth rate increases with stress and with crack length; (*c*) cracks initiate sooner in notched specimens than in unnotched specimens, for the same fatigue life; and (*d*) there is much more scatter in the crack initiation stage than in the propagation stage. It may be seen that these observations are all intimately related to one another metallurgically and mechanistically, but that they fall far short of providing a reasonable understanding of the phenomena involved.

Several recent investigations (4,5,6) have reported that sheet specimens containing a central stress concentration exhibited fatigue cracks, the plane of which was initially perpendicular to the plane of the sheet. At some undefined later stage of fatigue life, the plane of the crack was reported to shift to an angle of 45 deg with respect to the specimen surface (5,6) and at times the crack plane shifted to two mutually perpendicular planes of maximum shear (4). Frost and Dugdale (4) and McEvily and Illg (5) reported that this transition did not seem to affect markedly the rate of growth of the crack. Martin and Sinclair (6), however, observed an increase in crack propagation rate associated with the shift of the crack plane to the plane of maximum shear.

Martin and Sinclair (6), using a fluctuating tension fatigue cycle, did not observe any "long hesitation periods" during the growth of fatigue cracks in 2024-T3 aluminum alloy. They indicate that such periods would be theoretically impossible under the given loading conditions, since only when a compressive stress is present would a crack close, and thus not act as a stress concentration.

Frost and Dugdale (4), on the other hand, show definite rest periods in their crack propagation curves for an aluminum alloy tested in a similar fashion. They state that "these curves clearly show an important feature which tends to be masked on the orthodox growth curves. Periods occur in the life during which the rate of growth decreases, sometimes quite abruptly and in some cases ceasing altogether, after which the growth continues at its original rate as though the 'dormant' period had never existed." They also observed that "the number of dormant periods and their duration in any one test appears to be absolutely random except that they are more prevalent and prolonged at the lower stresses (as would be expected)." Frost and Dugdale exhibit their concern for these deviations by stating that "clearly, any law of continuous growth deduced from data obtained during this phase would not give a satisfactory representation of the process." They checked their specimens using metallurgical and microhardness tests and were unable to reveal any peculiarities immediately ahead of the dormant crack. They point out, as well, that this almost certainly could not be a fortuitous circumstance, since both cracks on any one specimen tended to be dormant at the same time.

It would seem that the preoccupation of these investigators and others with obtaining data that will smoothly and continuously fit a monotonic function of several variables is an oversimplified empirical approach to the problem. Since crack growth is generally accepted as being a discontinuous, nonlinear process, it might be suggested that much could be learned from a study of the discontinuities and nonlinearities. Head's theory for the growth of fatigue cracks (7) as well as those theories due to Weibull (8), McEvily and Illg (5) and Orowan (9) take these facts into consideration.

Cummings and coworkers (10) have stated that beyond an erratic preliminary stage of crack growth (0.010 in.) the further propagation of the crack "is characterized by continuous, smooth growth with relatively little variability." These investigators also report, for SAE 4340 steel heat-treated to a wide range of tensile strengths and tested in rotating bending over a range of stress and cyclic frequency, that the number of cycles required to form a crack 0.010 in. long is about 55 to 65 per cent of the total life of any specimen.

Hunter and Fricke (11) have studied crack initiation and propagation on notched specimens of 6061-T6 aluminum alloy tested in rotating bending. They periodically stopped their tests and made replicas of the notch structure for microscopic examination. They were able to show smooth curves for crack initiation similar in shape to the *S-N* curve and existing below the "notch endurance limit." They found crack initiation to be strongly influenced by notch severity. They reported that cracks quickly reached some critical size and then often stopped growing for an appreciable period. If the test stress was greater than the notch endurance limit the crack was later seen to resume growth "at the original rate." At lower stresses the cracks stopped growing altogether when they had attained critical size. These authors, however, found that hesitation periods at higher stresses were of such short duration as to be relatively undetectable. They also reported that the length of the hesitation periods was greater at lower stresses.

It should be noted that the data reported above were obtained in several fashions. Cummings, et al (10) measured tip-to-tip crack length every thousand cycles. McEvily and Illg (5) recorded cycles required for 0.10-in. incremental advances in crack length, as did Frost and Dugdale (4). Martin and Sinclair (6), however, optically measured crack length while testing at 600 cpm. Their data are tabulated as crack length at a given number of cycles; and using this method they were able, at times, to report crack lengths every two or three cycles during the final stages of propagation. It was not stated exactly how this could be accomplished.

Finally, only a single set of observations are known to exist which clearly show anisotropy associated with crack propagation. Lipsitt, et al (12) examined the fracture surfaces of rotating beam fatigue specimens of 2024-T4 aluminum alloy. They reported that fractures of transverse specimens taken from an extruded wing spar showed the area of final fracture to be elliptical in shape, with one end of the major axis of the ellipse at the position on the fracture cross-section where the fibers were shortest. These and other observations suggested that the crack had initiated at a point where the fiber length was shortest and that the rate of crack propagation was considerably faster in the fiber direction. On the other hand, their observations of the fractured surfaces of longitudinal specimens showed no anisotropy of propagation; the area of final fracture was circular.

Two further observations from that investigation will be of import in the later discussion: (1) It was observed that the area of final fracture was smaller for the longitudinal specimens than for the transverse specimens at a given stress, indicating that the cracks in longitudinal specimens tended to propagate further than did those in transverse specimens before fracture. (2) In this same vein, it was shown that the effectiveness of the notch was more pronounced at higher stresses; the fatigue strength reduction factor decreased continuously as life increased. The method of examination used in the above reported investigation was not suited to discerning any possible anisotropy (in life) associated with the formation of a crack.

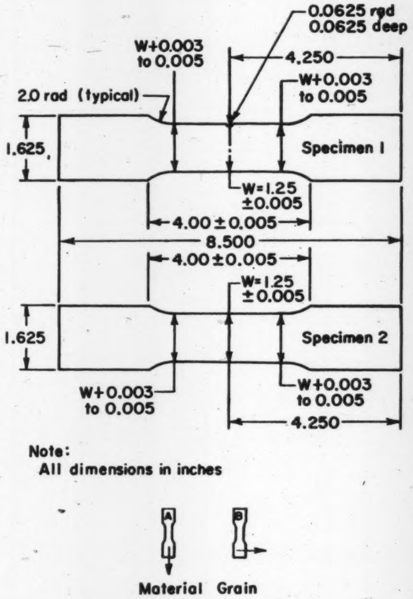


FIG. 1.—Experimental Specimen.

EXPERIMENTAL WORK

The specimens used in this investigation were prepared from 1100-H18 aluminum alloy.[3] The material, received in the form of sheet 0.051-in. thick, had been especially chosen from a nearly inclusion-free lot. Microscopic examination revealed the effects of cold working this alloy to the full-hard condition—the grains were elongated in the rolling direction and flattened in the transverse direction. The grains were about 0.007 to 0.014 in. long (ASTM 0–2) and 0.0009 to 0.0018 in. wide (ASTM 6–8).[4] The hardness of the material was Diamond Pyramid Number (DPN) 52 measured with a 10-kg load.

The geometry of the unnotched tensile specimens is shown (as specimen 2) in Fig. 1. The tensile properties were obtained on an Instron testing machine using type A-1, SR-4 strain gages mounted back-to-back on opposite sides of the specimens. The average of three tests gave the yield strength at 0.2 per cent offset as 24,000 psi, and the ultimate tensile strength as 27,500 psi. The tensile properties of this material were apparently independent of grain orientation. It should be noted that this material exhibited better than average tensile properties for this type of alloy.

The geometry of the notched fatigue specimen is also shown (as specimen 1) in Fig. 1. The notch selected imposes only a very mild stress concentration; its main purpose was merely to localize the point of crack initiation. A single notch was chosen because it was desired to follow the propagation of a single crack as far as possible prior to failure of the specimen, and because it was desired to have the photographic equipment as near the specimen as possible to obtain a large image.

The specimens were prepared by stacking specimen blanks in a milling machine and machining them in groups. The final milling cuts were limited to 0.003 in.

---

[3] Furnished through the courtesy of the Aluminum Company of America.

[4] Tentative Methods for Estimating the Average Grain Size of Metals (E 112 – 58 T), 1958 Book of ASTM Standards, Part 3, p. 506.

Specimen surfaces were protected during machining to prevent scratching: the notches were not polished after machining.

A total of 30 specimens have been tested to date; 15 of which had longitudinal grain (designated as A) and 15 had transverse grain (designated as B). The tests were performed at seven stress levels in the range of 13,000 to 24,000 psi. These stresses were chosen so that the total fatigue life rarely exceeded 50,000 cycles. This procedure was necessary because of the nature of the testing machine employed, its range of cyclic frequencies, and the need to synchronize accurately the stop-motion photographic equipment to the peak of the stress cycle.

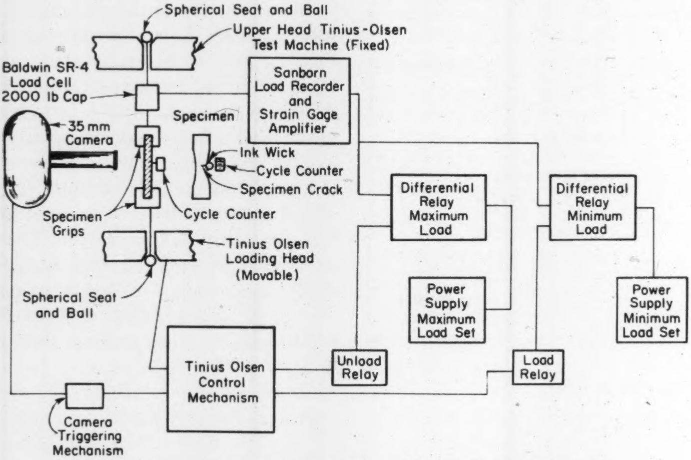

Spherical Seat and Ball
Upper Head Tinius-Olsen Test Machine (Fixed)
Baldwin SR-4 Load Cell 2000 lb Cap
Sanborn Load Recorder and Strain Gage Amplifier
35 mm Camera
Specimen
Ink Wick
Cycle Counter
Specimen Crack
Differential Relay Maximum Load
Differential Relay Minimum Load
Specimen Grips
Cycle Counter
Tinius Olsen Loading Head (Movable)
Spherical Seat and Ball
Power Supply Maximum Load Set
Power Supply Minimum Load Set
Tinius Olsen Control Mechanism
Unload Relay
Load Relay
Camera Triggering Mechanism

FIG. 2.—Experimental Setup for Tinius Olsen Testing Machine.

The fatigue tests were performed in a mechanically driven Tinius Olsen universal testing machine, modified to allow automatic cyclic loading in tension. A block diagram of the experimental equipment is shown in Fig. 2. It can be seen that the modifications were extensive, but for the sake of brevity they will not be described in detail. It should be noted that during testing the cracks were made more visible through the use of a wick saturated with nondrying ink and fastened to the specimen so that the ink flowed into the propagating crack.

The cycling was accomplished between limits of load, with a constant minimum load that produced a nominal stress of approximately 1200 psi; the cyclic frequency was 8 cpm. The stress calculations were based on the net area of the specimen at the notch, and the stress concentration of the notch was not considered.

The propagation of the cracks during testing was observed visually with a low-power microscope while the camera was photographing the specimen and a cycle counter simultaneously. The resulting films were analyzed on a microfilm viewer at approximately 12 magnifications. The crack lengths were measured from the root of the notch using a variable Gerber scale. The scale of measurement was determined for each test by photographing a machinist's scale held in the plane of the specimen. The Gerber scale was adjusted prior to analyzing each film so that one Gerber unit was equal to 0.005 in. It was possible to read one-half Gerber unit, or 0.0025 in. It is estimated that the accuracy of any individual measurement is of the order of one-half Gerber unit, and no minute crack jumps were recorded unless they were at least 0.0025 in. in length. A line with a finite slope is used in Figs. 3, 4, 6, and 7 to show a succession of very small increases in crack length.

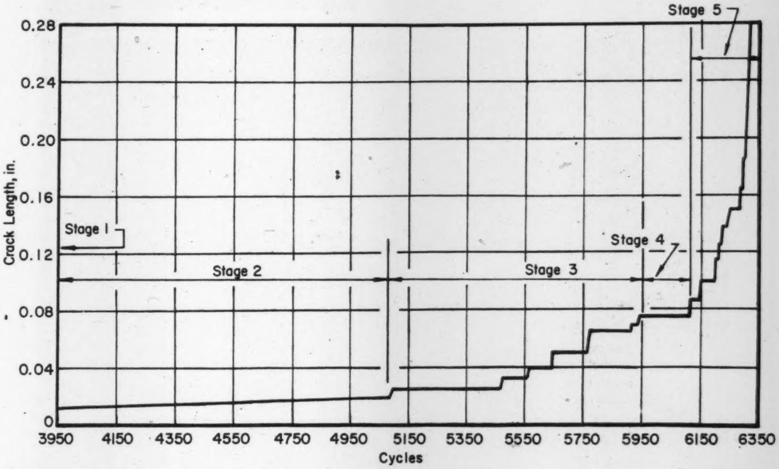


FIG. 3.—Crack Length *versus* Number of Cycles. Specimen NSB → 3. Maximum Stress 20,000 psi.

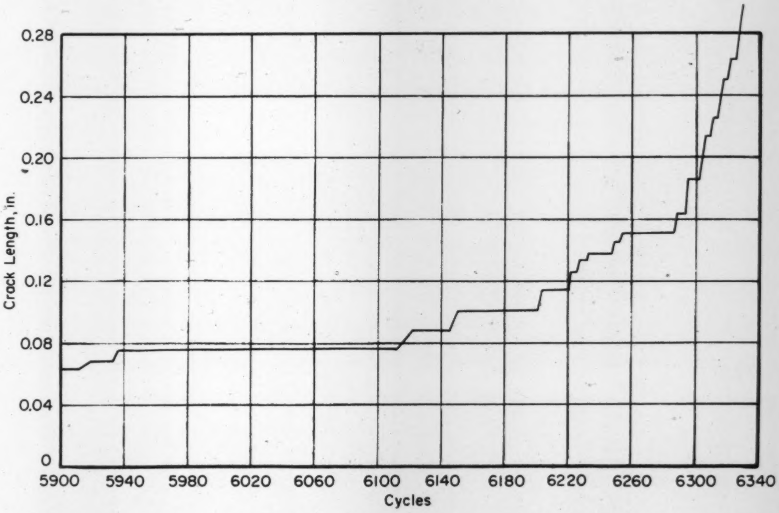
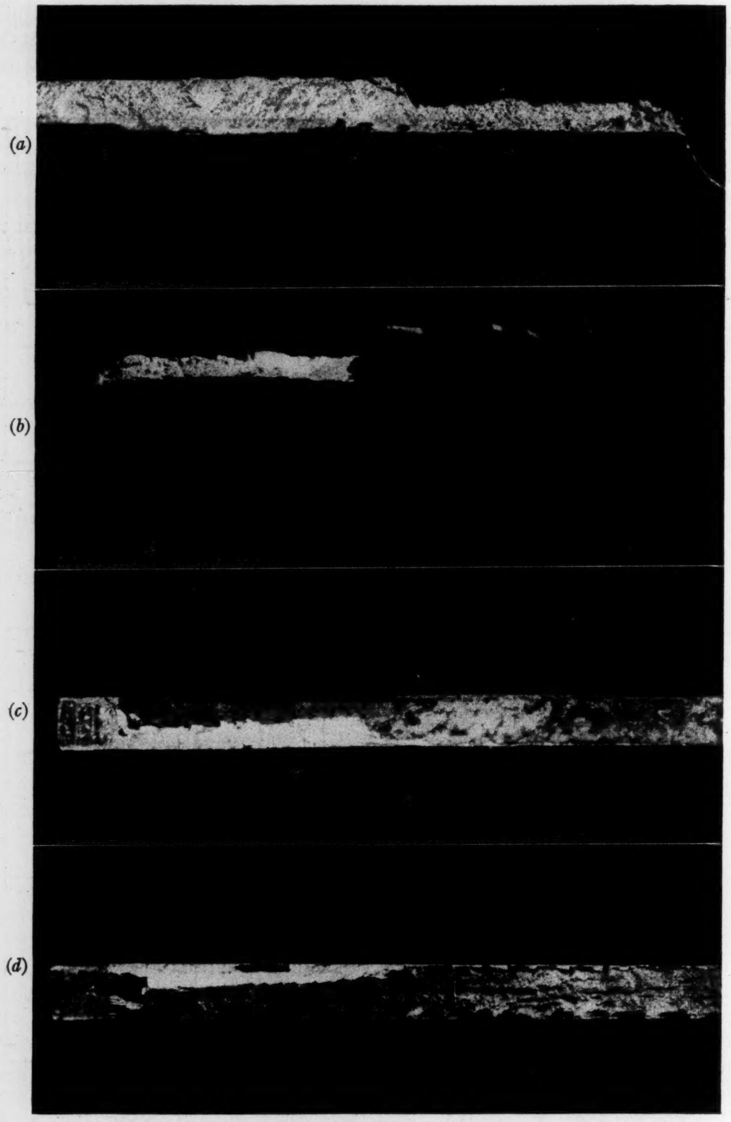


FIG. 4.—Terminal Crack Length *versus* Number of Cycles. Specimen NSB → 3. Maximum Stress 20,000 psi.



(*a*) and (*b*)—Two side views of a single specimen showing regions of single and double shear (approximately ×5).

(*c*) and (*d*)—Views of two specimens showing top views of crack topography. Specimen (*c*) is same as (*a*) and (*b*) (approximately ×5).

FIG. 5.—Typical Crack Topography.

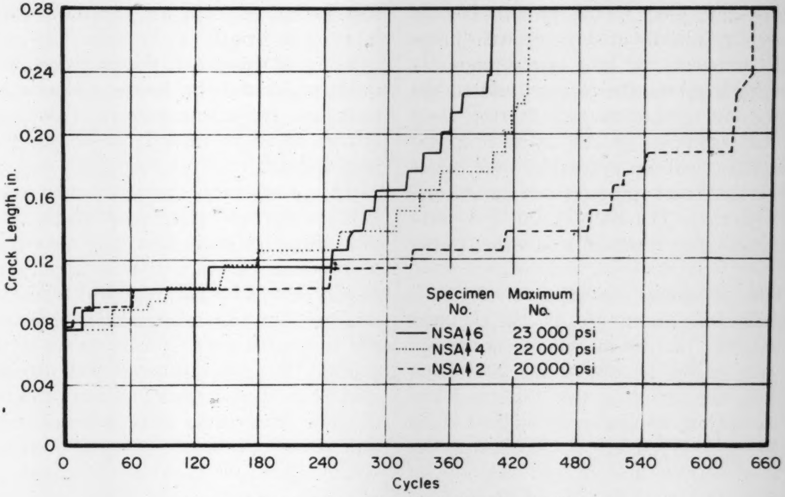


FIG. 6.—Crack Propagation as a Function of Stress.

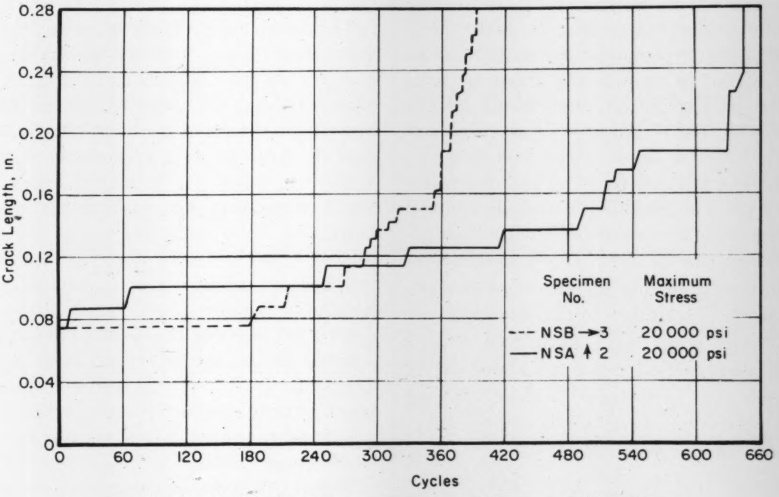


FIG. 7.—Crack Propagation as a Function of Specimen Orientation.

RESULTS AND DISCUSSION

An example of a typical crack propagation curve is shown in Figs. 3 and 4. Figure 3 shows a general picture of the entire curve, while Fig. 4 shows the right-hand portion of this curve on an expanded scale. It can be seen that the propagation of a fatigue crack is neither linear nor continuous. These curves do not approach linearity even on a semilogarithmic plot. Crack growth is seen to consist initially of short growth jumps and comparatively long rest periods. As the crack grows the jumps tend to get larger and the rest periods shorter. Only typical examples of the crack propagation data will be presented here, since all of the crack propagation curves are very similar. The bulk of the *S-N* data obtained are presented in another paper (13).

The foregoing description of crack propagation seems to fit the current qualitative picture of this phenomenon. It can easily be seen that hesitation periods are certainly the rule, and not an exception, as seemingly suggested by Frost and Dugdale (4). Similarly, the lack of observation of rest periods by Martin and Sinclair (6) and Cummings and co-workers (10) can only be due to the use of an observational frequency that was low enough to smooth the data.

The crack propagation curve may be considered to contain five stages as indicated in Fig. 3. An analysis of all the available data indicates that these periods may be summarized as follows:

1. The fatigue crack nucleus originates very early in fatigue life and slowly and discontinuously enlarges until it has attained a size of about 0.005 to 0.015 in. Our measurements do not allow a more accurate description of this nucleus.

2. The crack then undergoes a long hesitation period equal to about 50 to 60 per cent of fatigue life depending on the perfection of the notch and the magnitude of the applied stress.

3. Next, the crack enlarges discontinuously at an intermediate rate until it attains a length of 0.075 to 0.125 in. This length is proportional to the magnitude of the applied stress.

4. The period of intermediate growth is followed by an abnormally long rest period (for this stage of growth). This rest period ends in an unusually large increase in length.

5. From this point, the propagation is quite rapid and the jumps and rest periods are irregular in nature. This stage is considered to extend to the point of final failure.

The present concept of the first stage is in qualitative agreement with the data of Cummings et al (10). The very long rest period of stage two is apparently that observed by Hunter and Fricke (11). The greatest amount of scatter in fatigue life is seen in the first two stages of the process. The general shapes of the growth curves are almost identical from the end of stage two; curves of both longitudinal and transverse specimens tested over a wide range of stress can be superimposed at the fracture points on the curves and are seen to be nearly identical during the last 400 to 500 cycles of propagation.

The five stages of growth can be related generally to the appearance of changes on the fracture surfaces. The plane of the crack is approximately normal to the direction of the applied stress during stage one. It is apparently during stage two that the crack changes its plane of propagation, and so the crack hesitates while this is being accomplished. Stage three, then, is seen as propagation along two mutually perpendicular shear planes which lie at 45 deg to the plane of the sheet. The fourth stage is similar to the second stage; here again energy is being used to change the plane of propagation. Thus, in stage five, the crack propagation has shifted onto a single shear plane.

Figure 5 illustrates the nature of these changes in the plane of propagation of the crack. Deviations from the topography just described generally consisted of (*a*) no visible normal portion of the crack, that is, it *began* along two shear planes or (*b*) the shear planes were not of the same width, that is, the intersection of the two planes was displaced from the center of the specimen.

This apparent relation of crack topography to the crack propagation curve is not as exact as one would hope it to be. Perhaps the major reason for this inexact relation is the fact that the crack propagation measurements were made on the surface of the specimen, whereas the topographic features are below the surface.

Several of these observations are in qualitative agreement with those reported by others (**4,5,6**). However, the transition during stage four onto a single plane at 45 deg to the load axis is definitely accompanied by an increase in growth rate, as observed by Martin and Sinclair (**6**), but in contrast to the observation of Frost and Dugdale (**4**). No immediate explanation is possible for these observations on the relation of propagation rate to crack topography; this research is continuing.

A comparison of several crack propagation curves for longitudinal specimens tested at various stresses, as in Fig. 6, clearly shows that the rate of growth of a crack (the slope at a given crack length) from any arbitrary length increases as the stress increases. Thus, at higher stresses the average rate of growth is greater than at lower stresses. There does not seem to be any effect of stress on final crack length for specimens of a given grain orientation. This is in contrast to the results of Lipsitt et al (**12**), who found that the area of crack propagation decreased with increasing stress. It is believed that the major cause behind the present observation is that the bending stresses in the specimen became quite large by the time the crack had grown to a length of 0.250 to 0.375 in., and that from this point onward the failure mechanism was not truly one of fatigue alone.

Figure 7, on the other hand, definitely shows an anisotropy of crack propagation rate with respect to grain direction in the specimens. The rate of growth of the crack from any arbitrary length was faster in the transverse specimens than in the longitudinal ones when tested at a constant stress amplitude. Further analysis of the data indicates that at any given number of cycles prior to failure, the crack in a transverse specimen was almost a constant 0.025 in. shorter than that in a longitudinal specimen. In the final 25 cycles, however, this relation breaks down and, in fact, the transverse crack becomes as long as that in a longitudinal specimen. The fracture photographs of Lipsitt et al (**12**), on the other hand, indicated that cracks in longitudinal specimens tended to propagate further than did those in transverse specimens prior to fracture. Here again it is believed that the complex state of stress imposed by the advanced fatigue conditions tends to mask any structural differences in the specimens.

The distinct and abrupt shift in the plane of crack propagation that was seen to occur in stages two and four in this analysis was also seen to precede a change in propagation velocity. A velocity change is to be anticipated following the first shift of the crack plane since the crack must now sweep out 40 per cent more area after the shift has occurred. First considerations would indicate, then, that the growth rate should decrease at this point, other things remaining equal. The growth rate, however, was seen to increase. This may be due to the fact that the initial propagation occurs in a (nominally) uniaxial stress field whereas propagation along the shear planes is occurring under a (nominally) biaxial state of stress.

The shift of the crack propagation plane in stage four is more difficult to comprehend. It is conceivable, however, that the loading conditions would cause

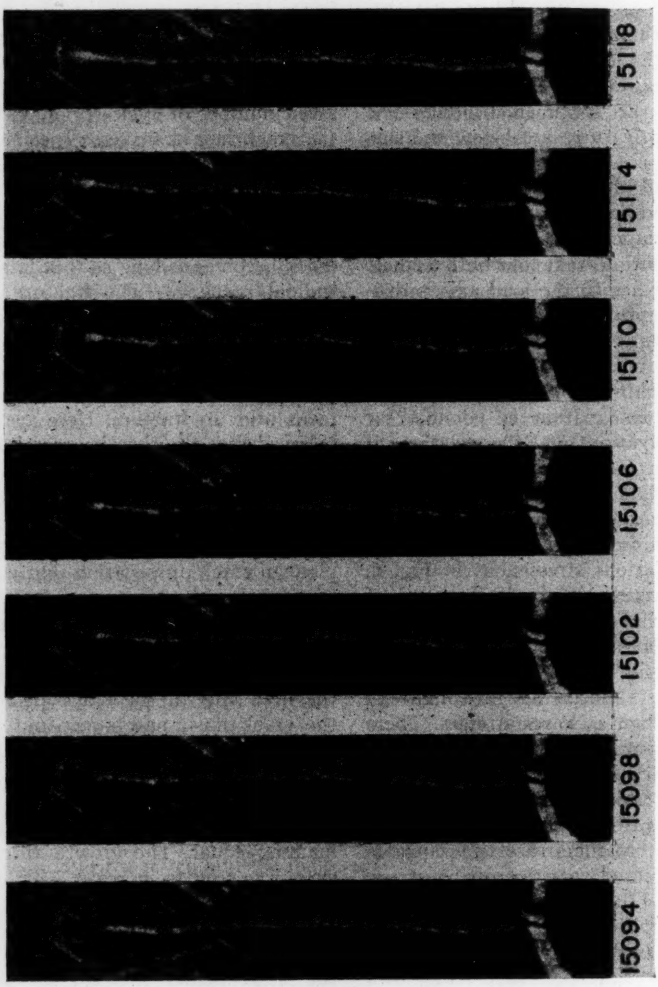

15118
15114
15110
15106
15102
15098
15094

FIG. 8.—Mechanism of Crack Propagation.

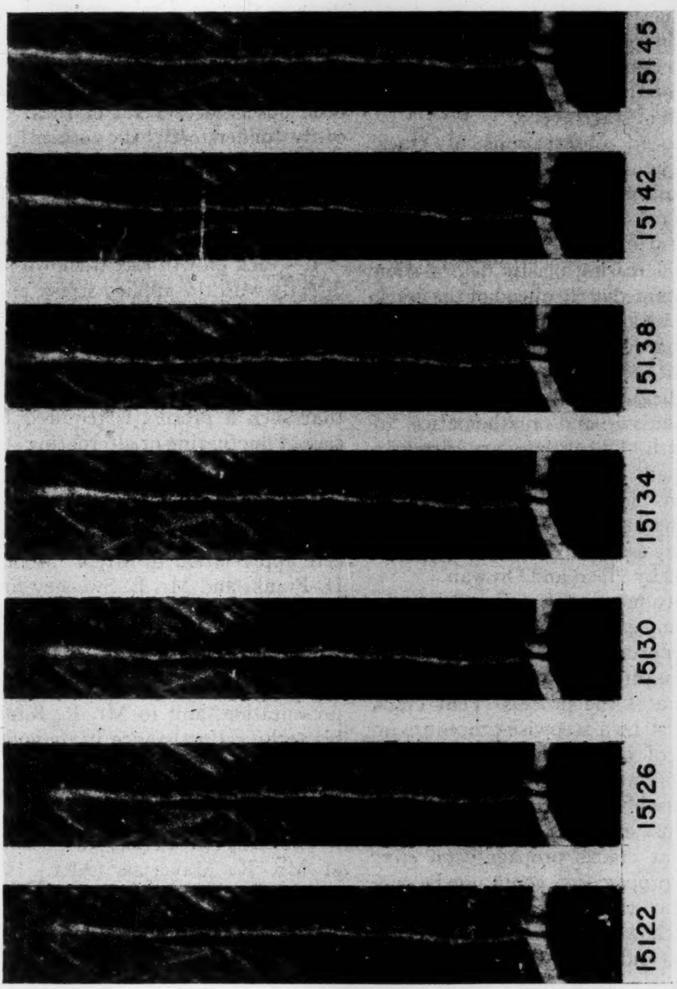

15145
15142
15138
15134
15130
15126
15122

FIG. 8.—*Concluded.*

a bending moment to be applied to the specimen, which would tend to shift the neutral axis of the unbroken area of the specimen toward the propagating crack. The introduction of the additional shear and normal stresses at the crack root due to this reaction may be associated with the pause of stage four and the accelerated growth of stage five.

The present observations of crack growth have also shed some light on the mechanism of propagation. Figure 8 shows several sequential photographs of an increment of crack growth. It can be seen that increasing plastic deformation occurring immediately ahead of the crack is followed by a crack length increase into the work-hardened area. This process is exactly that described by Orowan (9) and used by Head (7) and others to discuss the mechanism and mathematics of crack growth. The authors are currently analyzing all the available data gathered in this investigation to determine whether they seem to obey the mathematical laws associated with the physical model used by Head and Orowan.

In this connection, it is important to note that any expression for crack growth which purports to be of other than an empirical nature must consider the stepwise nature of the process. That crack growth must be a stepwise procedure on any scale of consideration in a plastic material follows directly from the (basically) sinusoidal nature of the applied stress. Thus, even if one were wont to assume that cracks propagate on *every* cycle, the propagation could not be continuous since the material about the tip of the crack work hardens as the crack progresses, and propagation could only occur at the peak of every cycle.

The present data qualitatively verify such an intermittent growth procedure. They further indicate that crack growth is a random walk process; this hypothesis is presently being investigated.

CONCLUSIONS

1. The growth of a fatigue crack is shown to be a discontinuous, nonlinear, stepwise process. Crack growth is seen to consist of short bursts of growth followed by periods of no measurable growth.
2. Five distinct stages of growth have been observed. Several of these can be easily understood; the others require further investigation.
3. The appearance of these stages is indicated on the fracture surface of the failed specimen.
4. Crack growth rate is shown to vary directly with the applied stress, and with the metallurgical structure of the material.
5. The stepwise nature of crack propagation is discussed and it is indicated that such a process is required for the case of fluctuating or alternating stresses.

Acknowledgments:

The authors wish to extend their sincere appreciation to Mr. E. Moore, Mr. D. Frank, and Mr. L. Sweeney for their assistance in carrying out the experimental program; to Miss A. Wells, Mr. R. Crowe, and Mr. F. Attinger for their assistance in preparing the data for presentation, and to Mr. R. Rautio for his technical assistance in designing the modifications of the test equipment, and to Mrs. Olga Hensler for typing the manuscript.

The interest, advice, and stimulation of Mr. R. Mayerjak (ARL), Mr. E. Callan (ARL), Mr. W. Eberhardt (ALCOA), Mr. L. Mayer (ALCOA), Mr. J. Poynter, Mr. C. A. Davies, Mr. W. Moreland and Mr. E. J. Hassell, of our laboratory, helped make this research possible.

The technical assistance of the Technical Photographic Laboratory is also gratefully acknowledged.

REFERENCES

(1) J. Schijve, "Fatigue Crack Propagation in Light Alloys," *NLL Report M2010*, Nat. Aeronautical Research Inst. (Holland), July 1956.

(2) L. J. Demer, "Review of Experimental Date on the Initiation and Propagation of Fatigue Cracks in Test Specimens," *WADC TR 55-86*, Part 2, June 1955.

(3) N. Thompson and N. J. Wadsworth, "Metal Fatigue," *Advances in Physics*, Vol. 7, No. 25, Jan. 1958, p. 72.

(4) N. E. Frost and D. S. Dugdale, "The Propagation of Fatigue Cracks in Sheet Specimens," *Journal Mechanics and Physics of Solids*, Vol. 6, p. 92 (1958).

(5) A. J. McEvily, Jr. and W. Illg, "The Rate of Fatigue Crack Propagation in Two Aluminum Alloys," *NACA TN 4394*, Sept. 1958.

(6) D. E. Martin and G. M. Sinclair, "Crack Propagation Under Repeated Loading," *Proceedings*, Third Nat. Congress Applied Mechanics, p. 595 (1959).

(7) A. K. Head, "The Growth of Fatigue Cracks," *Philosophical Magazine*, Vol. 44, p. 925 (1953), and *Journal of Applied Mechanics*, Vol. 78, p. 407 (1956).

(8) W. Weibull, "Effect of Crack Length and Stress Amplitude on Growth of Fatigue Cracks," *FFA Report 65*, Aeronautical Research Inst. (Sweden), Feb. 1956.

(9) E. Orowan, "Theory of the Fatigue of Metals," *Proceedings*, Royal Soc. (London), Vol. 171, Series A, p. 79 (1939).

(10) H. N. Cummings, F. B. Stulen, and W. C. Schulte, "Research on Ferrous Materials Fatigue," *WADC TR 58-43*, Aug. 1958.

(11) M. S. Hunter and Wm. G. Fricke, Jr., "Cracking of Notch Fatigue Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 643 (1957).

(12) H. A. Lipsitt, G. E. Dieter, G. T. Horne, and R. F. Mehl, "Study of Effects of Microstructure and Anisotropy on Fatigue of 24S-T4 Aluminum Alloy," *NACA TN 3380*, March 1955.

(13) R. B. Baird, F. W. Forbes, and H. A. Lipsitt, "Tensile and Fatigue Properties of Laminate Sheet Structures," see, p. 755, this publication.

DISCUSSION

Mr. P. R. Toolin[1] (*presented in written form*).—Hesitations and changes in the rate of propagation of fatigue cracks appear to be fairly well established. As the accompanying Fig. 9 shows, we have also noted these over a wide range of nominal stresses. This figure shows the growth of fatigue cracks started from small holes drilled in R. R. Moore specimens of hot rolled SAE 1015 steel. This steel had a fatigue limit of 27,500 psi and an average grain diameter of 0.001 in. Hesitations can be seen to occur all the way from stresses that are well up in the plastic range down to tests that result in nonpropagating cracks.

However, one wonders if these hesitations are not greatly influenced by the fact that commercial metals are composed of finite grains, variously oriented, and are certainly neither completely homogeneous nor isotropic. In a 0.3-in. thick plate specimen of alloy steel under zero-to-maximum stressing, with a crack growing at an average rate of about 1×10^{-5} in. per cycle, periods of complete dormancy at the surface did not appear to exceed 100 cycles in duration. In a similar specimen of hot rolled steel under zero to 32,000 psi stress, these periods were not observed to last over 500 cycles. Of course, is it not possible that while the crack has momentarily slowed at the surface, it may be progressing inside the specimen? Is it not possible that the character of the hesitations is influenced by the number of grains and other inhomogeneities across the crack front?

In regard to the relative propagation rate in the longitudinal and transverse specimens, our work, as does the authors', indicates that the propagation rate is influenced by stress. Curve one in our Fig. 10 was derived from the tests shown in Fig. 9. The growth rate was averaged over the period of crack-length growth from 0.015 to 0.105 in., because here the influence of the starting hole has diminished and the nominal stress is not changing rapidly. That this is not peculiar to the particular geometry of R. R. Moore specimens is shown by Fig. 11. Here again the propagation rate increases as the nominal stress is increased in these 0.3-in. thick plate specimens. Our work has also shown that the propagation rate is influenced by the stress concentration variations attending the changing crack length in these plate specimens. It thus seems that the growth rate is influenced by the actual stress and strain conditions at the crack front. As the fatigue strength taken transversely is normally lower than that taken longitudinally, the transverse specimens, at a given nominal stress, are operating higher on their *S-N* curve than are the longitudinal ones. Thus from Figs. 10 and 11 it might be predicted that the cracks in the transverse specimens should propagate at a higher rate for the same nominal stress.

Mr. H. T. Corten.[2]—Because the crack propagated from one side of the

---

[1] Mechanics Department, Westinghouse Research Laboratories, Pittsburgh, Pa.

[2] Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

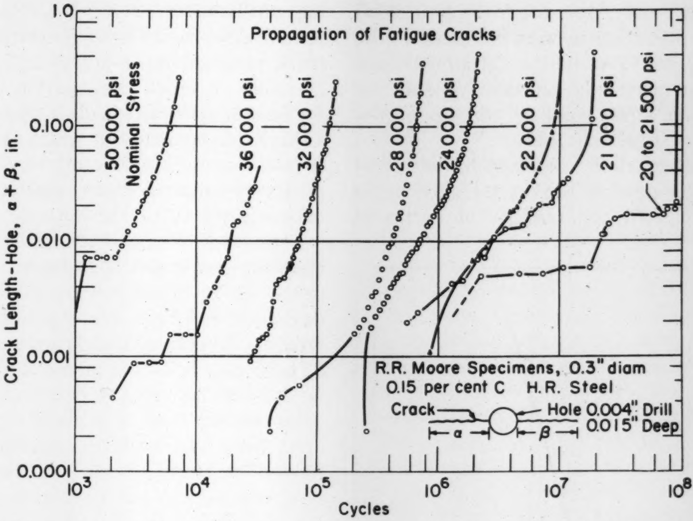


FIG. 9.—Growth Curves for Fatigue Cracks, Started at Small Holes, in R. R. Moore Specimens of Hot-Rolled Low-Carbon Steel.

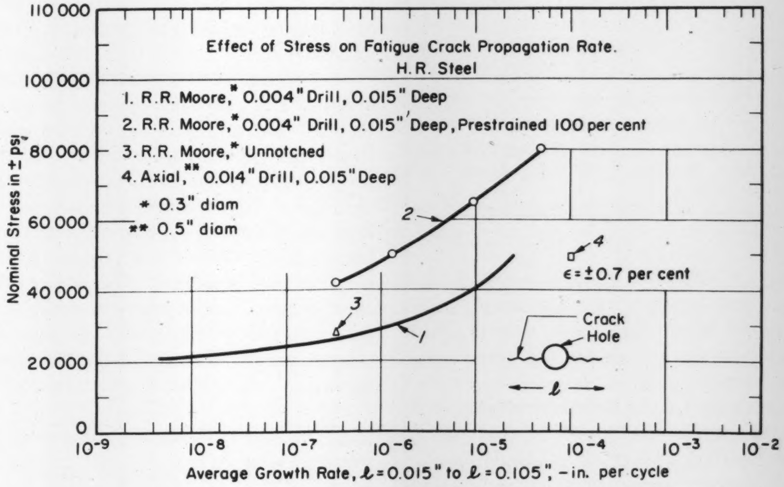


FIG. 10.—Influence of Nominal Stress on Fatigue Crack Growth Rate. Curve 1 is derived, in part, from the curves of Fig. 9.

specimen only, the eccentricity of load on the uncracked section increased as the crack grew. How did the authors account for the increasing eccentricity and the changing stress field at the tip of the crack as it propagated?

In other studies reported in the literature, the logarithm of crack length varies linearly with the number of cycles of load, N, when the crack length is less than one tenth of the specimen width. Beyond this length, these studies show that the linear relationship on a semilogarithmic diagram is no longer valid. It appears that this relationship may be valid for the present data also, within the restriction on crack length. Obviously in the range where the crack is from one fourth to one half of the width of the specimen, the rate of crack propagation is increasing very rapidly.

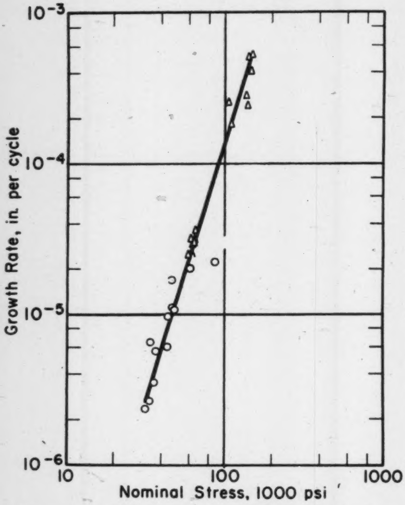


FIG. 11.—Influence of Nominal, Zero-to-Maximum Bending Stress on Fatigue Crack Growth Rate.

The alloy steel, wide plate specimens were 0.3 in. thick.

Finally, I am curious about the changing mechanisms of crack propagation. Specifically, from visual observations, is crack propagation in stages 2, 3, 4, and 5 similar to crack propagation in long-life fatigue specimens or is it more nearly a static phenomenon in which the crack becomes unstable and jumps rapidly for a relatively large distance, similar to the failure in the static tension test?

MR. R. E. PETERSON.[3]—The authors mention that oyster shell marks are evidence of a discontinuous fatigue behavior. It has been our experience with ordinary laboratory tests, run at constant deflection or constant moment, that you do not usually get oyster shell marks. In other words, these are found in service and there you have discontinuous behavior — stopping and starting, and changing of the load. This is a minor point, since the intermittent crack growth described by the authors does not require additional substantiation. In general, I think the paper is very interesting and certainly we have to think of fatigue cracks propagating intermittently.

MR. WILLIAM G. FRICKE, JR.[4] (*by letter*).—The authors are to be commended for an excellent job in using time-lapse photography to record the propagation of fatigue cracks. The advantage of their method is, of course, the high frequency with which observations can be made. Would the authors care to comment on how their observation that the propagating crack started and stopped many times for appreciable periods might be reconciled with the observations of Ryder[5] that a striation is formed on the fracture surface for each cycle of stress? Ryder's work implies that the crack propagates at least a little way each cycle.

---

[3] Manager, Mechanics Department, Westinghouse Electric Corporation, Research Laboratories, Pittsburgh, Pa.

[4] Research Metallurgist, Metallography Division, Aluminum Company of America, Alcoa Research Laboratories, New Kensington, Pa.

[5] D. A. Ryder, "Some Quantitative Information Obtained from the Examination of Fatigue Fracture Surfaces," *RAE Technical Note, Met. 288*, Royal Aircraft Establishment, Sept., 1958.

Concerning the nondrying ink which the authors used to make the crack more visible, have the authors any indication of the sensitivity of the method? Experience at the Alcoa Research Laboratories indicates that very small fatigue cracks, which are readily detectable by a microscope, are not disclosed by a dye penetrant. In Fig. 8 of the paper, are the dark areas which narrow in width toward the tip of the crack, portions of the specimen that have a different contour from the original surface or are they merely areas covered by ink?

In comparing the results of this paper with previous results of Hunter and Fricke discussed in the paper, it is necessary to bear in mind that the methods of testing and observation differed in the two cases. The present authors used axially-loaded sheet-type specimens and were looking at fatigue cracks growing away from a notch. In the work of Hunter and Fricke, notched rotating-beam specimens were used and the cracks were observed growing along the base of a notch. Effectively, the directions of observation in the two cases were at right angles to each other.

It seems probable, however, as the authors stated, that the hesitation period observed by them in the second stage of crack growth is comparable to the hesitation period observed by Hunter and Fricke. A problem arises, however, in interpreting the other stages. While the intermittent starting and stopping observed by the authors has also been observed at Alcoa Research Laboratories, no consistent second hesitation period (the authors' fourth stage) has been detected.

There is some question as to the authors' explanation of the five stages. According to them, a long hesitation period is needed to change from a cracking plane normal to the stress to two planes at 45 deg to the stress. Yet this hesitation period is presumably comparable to Hunter and Fricke's, where cylindrical specimens were tested and the crack plane was always nominally perpendicular to the stress.

The authors observed a slight increase in rate of crack growth at the end of the first hesitation period and ascribed the increase to the presence of biaxial stresses. This explanation is open to some question because biaxial stresses (although perhaps of a different type) have been present since the beginning of the test, due to the presence of the notch. During this hesitation period, the crack is still within the stress field of the notch.

The second hesitation period (the fourth stage) presumably results from the time needed to change from a double-shear to a single-shear type of deformation. It would be helpful if the authors showed a photograph of a fracture surface demonstrating the changing character from a normal, to a doubly inclined, to a singly inclined surface. Because the length of the crack is known for each of the stages, the photograph could readily be marked with the locus of the crack tip at the various periods.

Assuming the existence of a second hesitation period, an explanation not advanced by the authors might be considered. At this stage, the crack is still comparatively short and the tip extends only one or two times the notch radius from the base of the notch. Perhaps, under these circumstances, the stress-amplifying power of the notch is no longer felt and the crack momentarily hesitates before going ahead on its own.

In describing the material used in their investigation as having been "especially chosen from a nearly inclusion-free lot" the authors have made an unfortunate error in terminology. The tiny particles present in the material are normal for 1100 alloy. These phases, properly referred to as constituents, are an integral part of the alloy and exhibit no harmful effects from a fatigue standpoint. This is in distinction to inclusions, which are extraneous, unintentional phases of a largely nonmetallic nature and which may be harmful.

In connection with anisotropy of fatigue crack propagation, some recent work at Alcoa Research Laboratories complements the present paper. The material under investigation is from the same lot previously reported upon in reference (12) of the authors' present paper. Rotating-beam specimens of a 2024-T4 extrusion are being tested with the axis of the specimen in the long transverse direction. The extrusion "fibers" of the material run perpendicular to the axis of the cylindrical specimens. We are finding in these specimens that the fatigue cracks tend to originate where the fibers are shortest. As the crack grows along the surface, it, therefore, changes from propagating in the longitudinal direction to the short transverse direction. We are finding that as the crack begins to propagate in the short transverse direction and cuts across the extrusion fibers, it grows more slowly than originally.

It must be admitted that any periodic observation of fatigue cracks tends to obscure the vagaries of their propagation and their frequent stopping and starting. The authors' method is noteworthy for disclosing these many details. From a practical standpoint, however, the effective over-all rate of propagation is a value of some interest. It is hoped that the authors will publish additional information on this subject.

MESSRS. ARTHUR J. MCEVILY, JR. AND WALTER ILLG[6] (*by letter*).—Prompted by the authors' interesting paper on fatigue crack propagation in the aluminum alloy 1100-H18, we have reviewed the data obtained in the course of similar work with the aluminum alloys 2024-T3 and 7075-T6.[7] We observed discontinuities in growth of fatigue cracks only for the combination of low cyclic frequencies (40 cpm) and high stress levels (40,000 psi). At higher frequencies (1800 cpm) and lower stress levels the crack growth appeared to be continuous. In this connection the work of Ryder[8] is of interest. Working with the British aluminum alloy L65, which is similar to 2024-T3, at a mean stress of 12,000 psi and a superimposed alternating stress of ±3000 psi, he observed finite growth of the fatigue crack with each application of the ±3000 psi stress. Such crack growth would appear to be continuous on a macroscopic scale.

The authors have divided the process of fatigue crack propagation into five stages, stage three being characterized by crack growth along two mutually perpendicular shear planes at 45 deg to the applied load, and stage five by growth along a single shear plane. Examination of our failed specimens revealed that after the initial period of growth during which the crack was normal to the applied load, the crack shifted at random to either two mutually perpendicular planes or one shear plane. In these tests internal notches were used so that two cracks formed. In some cases in a single specimen one crack would shift to two planes while the other shifted to one. Once the shift had been made the crack usually grew without further shift of plane or planes until fracture occurred. In further contrast with the authors' findings, no discontinuities of growth were noted during the transition period. This may have been due to the fact that the transition from the normal plane to a shear plane took place gradually as the crack increased in length. In our tests then, two, rather than five, stages of crack growth were associated with fatigue crack propagation.

---

[6] Aeronautical Research Engineers, NASA, Langley Research Center, Langley Field, Va.

[7] Arthur J. McEvily, Jr., and Walter Illg, "The Rate of Fatigue Crack Propagation in Two Aluminum Alloys," *NACA TN 4394*, Nat. Advisory Committee for Aeronautics (1958).

[8] P. J. E. Forsyth, D. A. Ryder, A. C. Smale and R. N. Wilson, "Some Further Results Obtained from the Microscopic Examination of Fatigue, Tensile, and Stress Corrosion Fracture Surfaces," *RAE Technical Note, Met. 312*, Royal Aircraft Establishment, Aug., 1959.

MESSRS. HARRY A. LIPSITT, F. W. FORBES, AND R. B. BAIRD (*authors' closure*).—We wish to thank Messrs. Toolin, Corten, Peterson, Fricke, and McEvily and Illg for their stimulating and interesting discussions.

In response to Mr. Toolin's questions we feel, as he does, that a great many of the vagaries of crack propagation are almost certainly due to the fact that we are dealing with real materials. We do not, however, know what contribution is being made by grain boundaries and the like. The grain size of our specimens was such that the grains were very much longer than they were wide. If a significant contribution was being made by grain boundaries we would expect to see different hesitation characteristics in the longitudinal and transverse tests. No such trend was observed.

We agree with Mr. Toolin's interpretation of our crack propagation data in longitudinal and transverse specimens. And we feel, as well, that our measurements of crack propagation along the surface may not be directly proportional to the interior movement of the crack. In this respect some recent research of Forsyth et al[8] has shown that only in the early stages of fatigue is the surface crack propagation at all proportional to that propagation which occurs within the specimen. However, for the static fatigue of hydrogen charged SAE 4340 steel Steigerwald et al[9] showed that crack growth in this case is definitely discontinuous internally. We have begun an experiment employing an electrical resistivity technique to ascertain if the discontinuities noted in our paper are simply surface phenomena or if these discontinuities occur internally, as well.

In answer to Mr. Corten's questions it should be noted that the end of the third stage of growth in our tests occurred when the crack length was 0.075 to 0.125 in. in length (crack length equals a maximum of $1/9\ W$). It has been indicated in the paper that we consider bending to play an important role in the growth of cracks from this point. Our data are also in agreement with the apparent straight line relationship noted for the early stages of growth. Specifically, a smooth curve through our stage two data is indicated as a reasonably straight line on a semi-logarithmic plot.

It is extremely difficult to relate the present observations of crack growth to that unstable growth occurring in a static tensile test. Even late in the fifth stage of growth our crack propagation curves indicate a maximum (surface) propagation of 0.015 to 0.020 in. per jump. It should also be remembered that the five stages of growth to which we refer are purported only to describe the present data. Continuing research in our Laboratory on symmetrical specimens of 5052-H34 show a similar but not identical pattern of crack progression. These most recent experiments show that the fatigue crack does not always exhibit the double shear facets whereas our unsymmetrical specimens all showed this behavior. It can only be hoped that continuing research in this area will explain these differences.

---

[9] E. A. Steigerwald, F. W. Schaller and A. R. Troiano, "Discontinuous Crack Growth in Hydrogenated Steel," *Transactions*, Am. Inst. Mining, Metallurgical, and Petroleum Engrs. (Metals Technology), Vol. 215, p. 1048.

Mr. Peterson's question is an interesting one. The fatigue literature does indicate, as he points out, that "oyster shell" markings are generally observed in service failures. However, these markings are sometimes observed in laboratory tests. I have observed them on some (but not all) specimens of iron and aluminum, but not on titanium. In this regard the photographs of Forsyth et al and Ryder clearly indicate (on a much finer scale than is here being discussed) that one striation results per cycle and further that the magnitude of the striations are proportioned to the peak stress of the cycle.

In response to Mr. Fricke's questions we do not believe that our results are in conflict with Ryder's. These results and those of Forsyth et al[9] are on vastly different scales of measurement, that is, the crack movements he has shown are of the order of a micron in length and thus the electron microscopic technique is about two orders of magnitude more sensitive than ours. We do not claim that the nondrying ink greatly increased our ability to locate a very small crack but rather that this ink successfully outlined an open crack so that the film density varied widely between cracked and uncracked portions of the specimen. The dark areas referred to on our Fig. 6 are merely areas covered by ink.

We are grateful for Mr. Fricke's further analysis of the five stages of growth which we observed and for the presentation of his most recent results on extruded 2024-T4. The fracture photographs reported and discussed in reference (12) (Lipsitt et al) of the present paper show the same crack growth behavior discussed by Mr. Fricke.

TENSILE AND FATIGUE PROPERTIES OF LAMINATE SHEET STRUCTURES*

BY R. B. BAIRD,[1] F. W. FORBES,[1] AND H. A. LIPSITT[1]

SYNOPSIS

Laminate sheet structures composed of a number of adhesive-bonded layers of 1100-H18 aluminum foil, with their directions of rolling oriented in various fashions with respect to one another, were prepared. The mechanical properties of these structures were determined and compared with the properties of solid sheets of the same material.

It was found that these structures have a smaller density than solid aluminum and that they exhibit substantially isotropic behavior in two orthogonal directions. On a strength-to-density basis these structures exhibit static tensile properties comparable to, or slightly higher than, those of a solid sheet. The tension failure is also different, involving less reduction of area and a definite reduction in the speed of final crack propagation. Under certain conditions the rapidly propagating crack stops completely before destroying the structure. Laminates of 1100-O foil fail by the very slow propagation of a crack and little reduction of area. In a fatigue test the laminate structures, at the same fatigue life as a solid sheet, show either a greater resistance to the formation of a crack or an ability to restrain crack propagation until the final stage of fatigue life.

One of the unfortunate aspects of fatigue is that the conditions that cause a crack to form are generally more than sufficient to provide for the eventual propagation of that crack to failure. Several research studies have indicated that crack initiation may, under the proper conditions, occur very early in fatigue life (**1–5**).[2] In the usual case, the crack, once formed, invariably continues to grow until failure occurs. However, it has been shown that in certain materials a crack may form but not necessarily propagate (**6**).[3] Strain aging and the fatigue limit phenomenon are usually involved in these situations. It may not always be necessary, however, to completely halt crack propagation; excellent use could be made of a material with an abnormally low rate of crack propagation.

It has been shown (**7,8**) that crack propagation in fatigue is structure-dependent; fatigue properties in the longitudinal direction in cold-worked materials are superior to those in the transverse direction. Thus, a definite advantage would obtain if high-strength materials could be produced with identical properties in two orthogonal directions in the plane of the sheet.

It was suggested by F. W. Forbes that

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Task Scientist, Engineer-in-Training of the Mechanics Research Branch, and Project Scientist of the Metallurgy Research Branch, respectively, Aeronautical Research Laboratory, Directorate of Laboratories, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] H. A. Lipsitt, unpublished research.

perhaps some of these desirable properties could be achieved by laminating cold-worked metal foils in the same manner as plywood. Laminates made in such a fashion should have the tensile properties of the foil providing the grain directions were properly oriented. Indeed, it might be possible that the initiation of a fatigue crack could be retarded because of the necessity of causing the crack to initiate in all the layers of a specimen. It was also conjectured that it would not be very serious if only a single layer of foil were cracked. Propagation of the crack might also be retarded because of the additional difficulty of individually moving a crack in each layer of the laminate structure.

To determine which, if any, of these possibilities had merit, several varieties of laminate sheet structures were prepared and their properties observed. In all cases the properties were compared with those of solid sheet.

The material properties thus far investigated are density, modulus of elasticity, yield strength, ultimate tensile strength, and fatigue behavior. Observations have also been made of the terminal crack propagation velocity in tension, annealing effects (prior to laminating), and ductility.

The available literature on this subject is very sparse; by far the greatest percentage of physical and mechanical property studies on laminates have been made on simple lap joints tested in shear. Some fatigue studies are currently in progress[4] but these are also being conducted on lap joints for the purpose of developing a meaningful design criterion for laminated joints.

Cross and co-workers (9) have reported the results of a very interesting and comprehensive study of the development of laminating techniques, the variables involved, and the properties of the resultant structures. They reported that, "If the area of the laminate was assumed to be only that of the metal plies, the calculated (mechanical property) results agreed with the actual test results." They concluded that the properties of laminated structures using FM-47 film adhesive were equivalent to those of solid specimens of equal metal area. Their fatigue results in reversed flexure were reported on a "net metal area" basis, and here they found no effect of variation in ply thickness or number of plies in the structure. They did find, however, that although the laminate structures showed more scatter than a solid sheet when tested in fatigue, the laminates did exhibit better fatigue life at lower stresses. They stated that this result may be partly due to the fact that one or more metal plies can be cracked and not affect the dynamic deflection in bending. Their fatigue specimens exhibited a fatigue crack in the outer ply early in the test, and they also found the fatigue crack to be discontinuous at the glue line, that is, the cracks were not continuous across the entire specimen test section.

EXPERIMENTAL WORK

The specimens used in this investigation were prepared from 1100-H18 aluminum alloy.[5] The material was received as 0.0035-in. thick foil from the same lot of aluminum used in a previously reported investigation (7). Microscopic examination of the foil revealed that the grains were elongated in the rolling direction; they were about 0.007 to 0.014 in. long (ASTM Nos. 0 to 2) and 0.0009 to 0.0018 in. wide (ASTM Nos. 6 to 8).[6]

The shape of the unnotched tensile specimen was the same as that previously used for solid sheet (7). The tensile properties were obtained on an Instron testing machine using type A-1, SR-4 strain gages mounted back-to-back on opposite sides of the specimens. These properties differed only slightly from those of the solid sheet specimens previously reported (7). The yield strength of the foil at 0.2 per cent offset was 24,250 psi and the ultimate tensile strength was 27,700 psi.

---

[4] W. J. Trapp, Wright Air Development Center, private communication.

[5] Furnished through the courtesy of the Aluminum Company of America.

[6] Tentative Method for Estimating the Average Grain Size of Metals (E 112 – 58), 1958 Book of ASTM Standards, Part 3, p. 506.

The laminate sheet structures were prepared[7] by spraying the surfaces to be bonded with FM-47 adhesive, stacking the foils in the desired grain orientations in a press, and curing for 30 min at a temperature of 325 F and a pressure of 200 psi. It had been determined that this treatment had a mildly deleterious effect on the mechanical properties of the individual foils. The significance of this will be discussed later in the paper.

Three grain orientations were used in producing the laminate structures. Several sheets were prepared in which the rolling directions of all layers were parallel. From these it was possible to obtain both longitudinal (designated as A) and transverse (designated as B) specimens. Other sheets (designated as L) were prepared in which the rolling direction varied 90 deg (as in plywood) between adjacent layers of foil. The third type of structure (designated as O) had the rolling directions of adjacent sheets angularly displaced by $\frac{180}{n - 1 \text{ deg}}$, where n is the number of plies in the laminate. The top and bottom plies of these laminates had their rolling directions parallel. All laminates were prepared to a thickness of about 0.052 in.

The A and B type laminates were mainly for control purposes. The number of specimens of these types prepared was only sufficient for the tensile and modulus determinations. Only specimens of the L and O type structures were subjected to complete examination under tensile and fatigue conditions.

The tensile properties of the laminate structures were determined in the same manner as for the foil and sheet (7). High-speed motion pictures (128 frames per sec) were made of the tensile failures and analyzed in the same fashion as the fatigue crack propagation films.

The modulus of elasticity of the various laminates was determined dynamically using dual-channel Brush and Sanborn strain-gage amplifying and recording systems. The modulus of elasticity under static conditions was determined with a portable Baldwin strain indicator. The load signal was obtained from the load cell of the Instron testing machine. Strain signals were obtained from two active type A-1, SR-4 strain gages, installed back-to-back on opposite sides of the specimens. The external bridge circuit was completed using two identical dummy gages. It is estimated that the over-all accuracy of the modulus determinations was of the order of ±2 per cent.

The shape of the fatigue specimen used in this investigation was identical to that used in the previous research (7). The fatigue testing equipment, the photographic techniques and instrumentation, and the methods of crack propagation analysis were identical to those used in a previous investigation. All fatigue tests were conducted in cyclic tension at a frequency of 8 cpm (7).

A total of 12 tensile and modulus specimens have been tested. Thirty-three fatigue specimens have been tested and their crack formation and propagation characteristics determined. The bulk of these were tested in the same range as used for the solid sheet (13,000 to 24,000 psi maximum stress per cycle).

---

[7] Through the courtesy of The Martin Co.

TEST RESULTS AND DISCUSSION

A summary of the elastic modulus and tension data for the laminated sheet structures is given in Table I.

In considering the modulus of elasticity of these structures it must be noted that laminates from sheet *X* contained only 12 metal plies, but were built up to the same over-all thickness as the 13-ply laminates (0.052 in.). Consideration of this fact does make it possible to account for the apparent modulus differences in the structures in a qualitative manner.

TABLE I.

Structure	Elastic Modulus, psi	Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent
Solid sheet...	10.00×10^6	24 000	27 500	2.3
Sheet *W*, grain *A*...	9.32	21 600	24 900	2.1
Sheet *W*, grain *B*....	9.41	21 500	26 900	2.1
Sheet *X*, grain *A*...	8.49	19 650	24 300	2.7
Sheet *X*, grain *B*....	8.75	22 800	26 000	2.5
Sheet *Y*, grain *L*....	9.39	21 800	25 900	2.1
Sheet *Z*, grain *O*....	9.40	21 950	25 900	2.4

Several tests, including the modulus measurements, have indicated that the adhesive in this statically indeterminate structure is, indeed, supporting a reasonable proportion of the applied load. The magnitude of this proportion will be indicated below, but it is interesting to note that this observation is in direct contrast to that of Cross and co-workers (9).

Using the modulus determinations it is possible to calculate an apparent elastic modulus for the FM-47 adhesive. This may be accomplished from considerations of load equilibrium and the assumption of strain equality required for the solution of redundant structures. With the above considerations it can be shown that

$$E_L = \frac{A_{AL}}{A_L} E_{AL} + \frac{A_R}{A_L} E_R \ldots\ldots (1)$$

where:

E = modulus of elasticity, psi

A = area of the structural layers, sq in.,

Subscript L = laminate,

Subscript AL = aluminum, and

Subscript R = adhesive.

This equation states that the modulus of elasticity of the laminate is equal to the sum of two terms, each of which is the product of the areal fraction of a given component of the structure and the elastic modulus of that component.

The results of this calculation indicate that the apparent elastic modulus of FM-47 in these structures varies from 1.84×10^6 to 5.05×10^6 psi, depending on the number of layers in the laminate, that is, on the thickness of the glue line as affected by the number of plies in the structure. It was found that the apparent elastic modulus of the adhesive was lowest in the 12-ply structures where the glue line is thickest. In the 12-ply structures the apparent modulus varies from 1.84×10^6 psi to 3.24×10^6 psi, for an average glue line thickness of 0.00091 in. In the 13-ply structures this variation was from 4.29×10^6 psi to 5.05×10^6 psi, where the average glue line thickness was 0.00054 in.

These data indicate a very marked dependency of the elastic behavior of the laminate structures on the thickness of the glue line. Clearly, a distinct advantage is to be gained for very thin glue lines, where the properties of the adhesive are apparently much greater than the bulk material would exhibit. The research of Cross et al in this field was limited to glue line thicknesses of 0.005 to 0.030 in.

(9). Their results make it apparent that in this range the properties of the adhesive are those of the bulk material and, as such, are distinctly inferior to those properties exhibited by a very thin layer of adhesive.

In this connection two problems are of immediate import and are presently under investigation in our laboratory. First, it is conceivable that much better properties could be obtained if the adhesive layer were to be made still thinner and secondly, it would be advantageous to any continuing study of these structures to know the "true" modulus of the adhesive as a function of its thickness.

Table I shows that, for the 13-layer structures, the yield strength at 0.2 per cent offset is of the order of 2000 psi less than for a solid sheet of the same alloy. The relation of ultimate tensile strengths is a little more favorable, the strengths of the 13-layer laminates being about 1500 psi less than for the solid sheet. It should be noted that the operation of curing the bonding adhesive required the laminates to be treated at 325 F for 30 min. This process reduced the hardness of the individual foils from about 52 diamond pyramid hardness (DPH) to about 47 DPH and lowered the strength of the foils to about 24,500 psi. Thus, it could not be expected that these structures would exhibit the maximum tensile properties of the untreated foil. However, this evidence indicates that structures prepared from foil materials whose strength would not degenerate during bonding could be distinctly superior to a solid sheet. An investigation to determine the merit of this hypothesis has begun in our laboratory.

In this regard it can be shown in another manner that the adhesive is contributing significantly to the strengths of the laminates. A mathematical analysis indicates that, if the metallic area alone were considered, the foils in the 13-layer structures would have to possess a yield strength of 24,700 psi and ultimate strength of 29,500 psi to exhibit the same gross laminate behavior as is observed. For the 12-layer laminate these strengths would have to be 24,100 and 29,900 psi, respectively.

One of the very striking properties of these structures is their ability to completely stop the propagation of the final failure in tension at constant strain rate. Sheet materials that are fully work-hardened by rolling generally fail by the formation of a deep-necked band in the plane of the sheet and at an angle of 45 deg to the tensile load. When the band has fully formed, and the load has fallen from its maximum value, a crack begins at one edge of the neck and very rapidly and suddenly propagates the full width of the sheet, causing failure.

The process of tension failure in the laminates is virtually the same as in the solid sheet, except that the necked region is not so deep as in the sheets, that is, the reduction of area is less, and consequently the load does not recede as much before the final fracture begins; also, the fracture plane is generally normal to the applied load. In several of the tests, however, these laminate structures were able to halt completely the progress of the final failure before the crack had traveled half the specimen width. In all these cases the load began to increase again and was seen to reach a peak before the failure continued, often as a slow, tearing failure. This inherent "fail-safe" behavior was observed on all the types of laminates studied, but not in all laminate tension failures. It has been ascertained from high-speed motion pictures of the tension failures that the velocity of final failure is considerably lower in the laminate structures. It has not yet been possible to analyze quantitatively this aspect of failure in laminated structures. It was thought initially that this "fail-safe" property might be associated with a restraint to bending afforded by the tensile sheet grips, but subsequent tests with pin-connected grips showed the same phenomenon.

Table II shows the density of the laminates and compares their tensile properties with those of solid sheet. The comparison here is made on a strength-to-density basis. It can be seen that the density of the 13-layer structures is about 7 per cent less than that of solid aluminum and that the 12-layer laminates are about 10 per cent less dense than aluminum. To calculate an apparent density for the adhesive in these structures it can be shown that the following formula is applicable

$$D_L = \frac{A_{AL}}{A_L} D_{AL} + \frac{A_R}{A_L} D_R \ldots\ldots (2)$$

Where the subscripts are as above (see Eq 1) and D is density, in pounds per cubic inch. This calculation shows the apparent density of the adhesive in sheets W, X, Y, and Z to be 0.0343, 0.0428, 0.0434, and 0.0369 lb per cu in., respectively. Since the density of the uncured liquid adhesive is 0.0355 lb per cu in., the apparent densities may be taken as being indicative of the perfection of the bonding layer, that is, the amount of entrapped air. Thus, the most consistent and the best behaved laminates might be expected to be those from sheet Y, with sheets X, Z, and W following in descending order. It is not possible at this time to completely evaluate this hypothesis; it can be seen, however, that the laminates from sheet Y show a smaller data scatter than the others.

TABLE II.

Structure	Density, lb per cu in.	Yield Strength / Density	Yield Strength Comparison, per cent	Ultimate Tensile Strength / Density	Ultimate Tensile Strength Comparison, per cent
Sheet	0.0980	2.45×10^5	...	2.75×10^5	...
Sheet *W*, grain *A*	0.0906	2.38	−2.8	2.75	0
Sheet *W*, grain *B*	0.0906	2.33	−4.9	2 99	+8.7
Sheet *X*, grain *A*	0.0879	2.24	−8.6	2.76	+0.4
Sheet *X*, grain *B*	0.0879	2.60	+6.1	2.96	+7.6
Sheet *Y*, grain *L*	0.0916	2.38	−2.8	2.83	+2.9
Sheet *Z*, grain *O*	0.0908	2.42	−1.2	2.86	+4

The comparison of properties on a strength-to-density basis indicates that the laminates are very similar to the solid sheets. The two more complex structures (L and O) seem to exhibit the least variability and are considered to be the most worthy of further consideration. Finally it must be remembered, in considering the impact of these comparisons, that the final properties of the foil from which the laminates were made are inferior to the properties of the sheet to which the laminates are here being compared. The variation of bond quality, indicated by the apparent adhesive densities, will also have an effect here.

Figure 1 presents the fatigue results gathered to date in this continuing program. It can be seen that in fluctuating tension the fatigue life of the L and O type laminates is very similar to that of the solid sheet for stresses below 20,000 psi. Above 20,000 psi, the fatigue life of the laminates decreases faster than that of the sheet. This is believed to be due to the rapid breakdown of the adhesive layer at the higher stresses. A slight tendency of the laminate structures to delaminate at the highest stresses was observed; this tendency was not observed at the lower stresses.

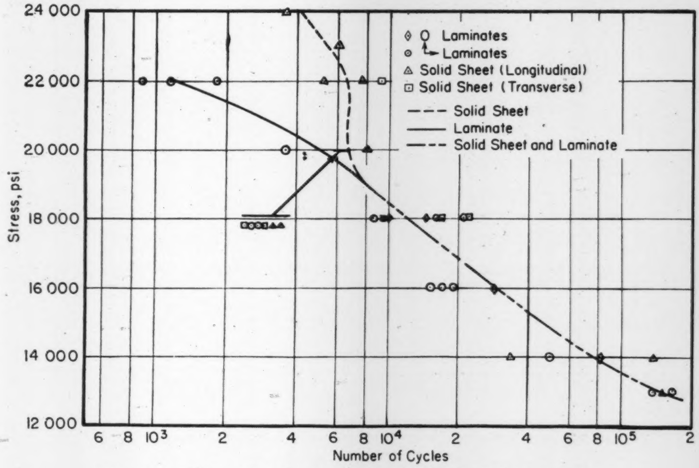


FIG. 1.—Fatigue Life Comparison on Nominal Stress Basis.

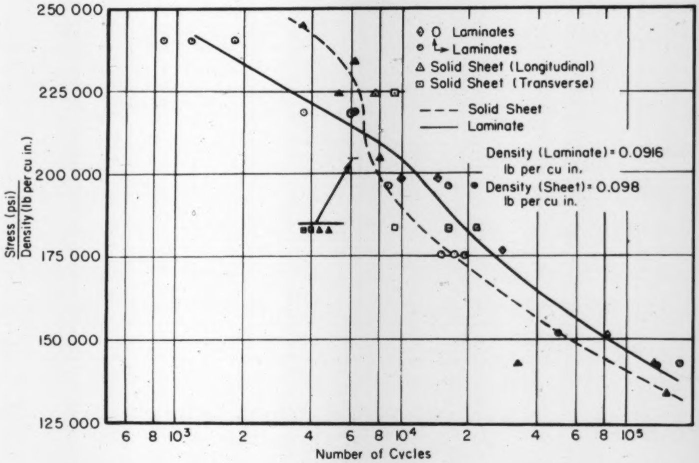


FIG. 2.—Fatigue Comparison on Nominal Stress to Density Basis.

Figure 2 is a fatigue behavior comparison between the laminated structures and solid sheet. Here the ordinate is a stress-to-density parameter to enable the various densities of these structures to be considered. Since the laminate structures are 7 to 10 per cent less dense than the sheet, the effect is to shift the laminate data vertically upward with respect to the solid sheet. This comparison indicates that at the lower stresses the laminate structures have fatigue properties superior to those of the solid sheet, at least where the density (or weight) of a structure is important. It would seem that the laminated structures might be increasingly more durable as the stress is decreased to levels of ordinary fatigue usage.

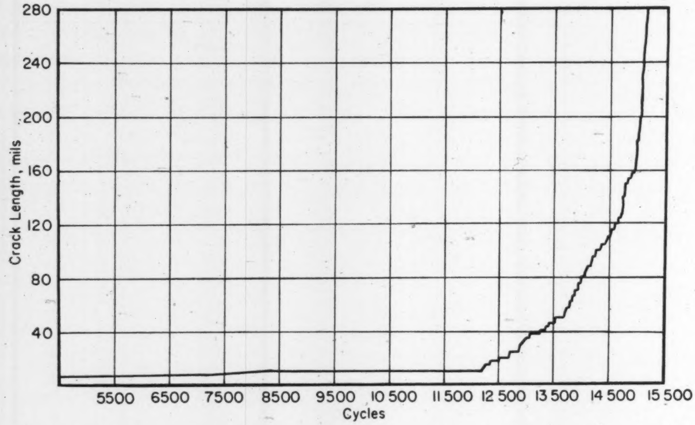


FIG. 3.—Crack Length *versus* Number of Cycles. Laminate NY ↑→ 10. Stress 16,000 psi to 1200 psi.

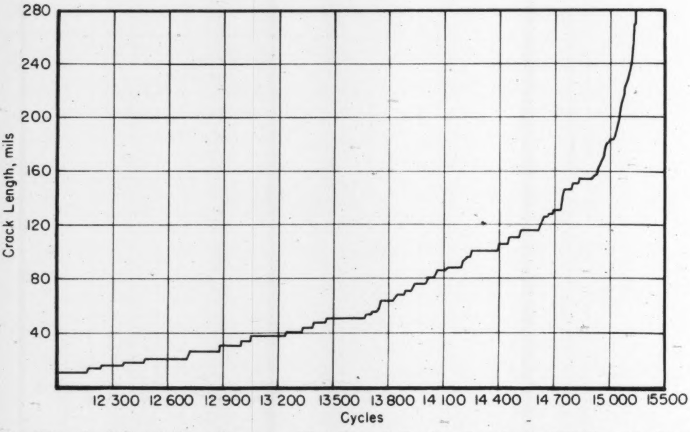


FIG. 4.—Terminal Crack Length *versus* Number of Cycles. Laminate NY ↑→ 10. Stress 16,000 psi to 1200 psi.

This research is now in progress in our laboratory. The present data are being extended to lower stresses; a portion of these data will be gathered in alternating bending at 1800 cpm to ascertain the effects of state of stress and speed of test.

Figure 3 shows a typical crack growth curve for these laminated structures.

Figure 4 presents the terminal portion of these data shown for clarity on an expanded scale. It can be seen that the general mode of crack growth in these structures is very similar to that previously reported for solid sheet (7).

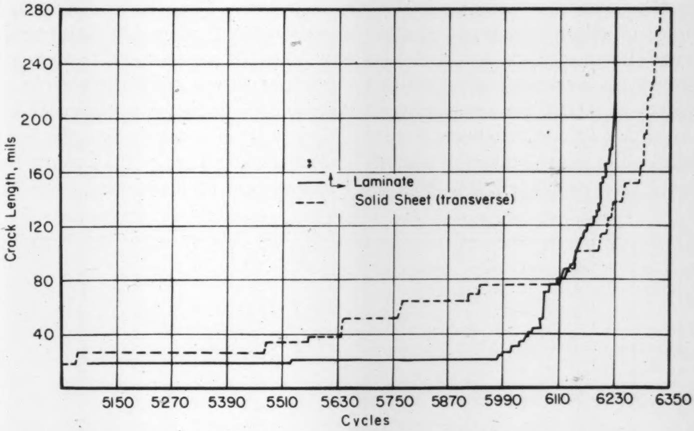


FIG. 5.—Crack Length *versus* Number of Cycles. NSB-3 Solid Sheet and NY ↑→ 5 Laminate Stress 20,000 psi to 1200 psi.

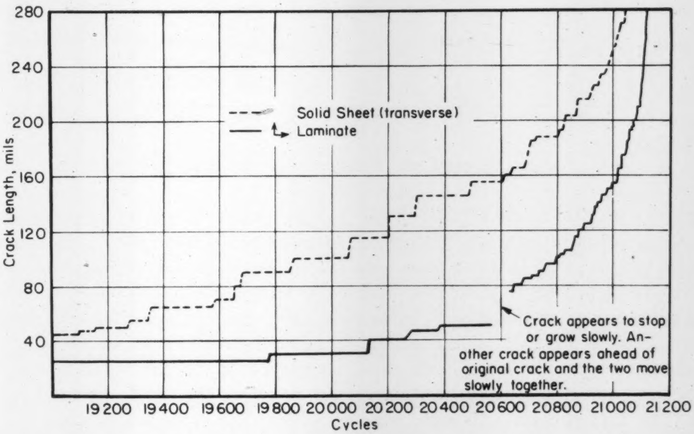


FIG. 6.—Crack Length *versus* Number of Cycles. NSB-22 Solid Sheet and NY ↑→ 7 Laminate. Stress 18,000 psi to 1200 psi.

Two differences in the fatigue behavior of the laminate structures have been observed. All of the solid sheet specimens previously discussed were observed to contain a crack very early in fatigue life. Several of the laminate specimens, on the other hand, did not exhibit fatigue cracks until about 25 to 30 per cent of the fatigue life had expired. Those laminate structures that did exhibit cracks early in life were able, however, to restrain the growth of the crack for a longer period than was the sheet.

Examples of these behavioral characteristics are shown in Figs. 5 and 6. Figure 5 shows an example of restrained crack growth at 20,000 psi comparing an L-type laminate to a solid sheet. Figure 6 indicates the same phenomenon at 18,000 psi. The laminated specimen in Fig. 6 did not exhibit a crack until about 1.5×10^4 cycles had been applied; some specimens tested at 16,000 psi have exhibited early appearance of cracks and exceptionally long rest periods before continued growth (Fig. 3) while others have exhibited a much later crack appearance and shorter initial hesitation periods. The fact remains, though, that regardless of the initiation time of the crack the laminated structures show an increased ability to slow the failure process. It is believed that this facet of laminate behavior is of greater import than the time required to initiate a crack, since crack initiation must be a strong function of notch perfection.

Figure 7 shows the variation in crack propagation rate as a function of the applied stress. These data clearly show that crack growth from any arbitrary length in identical specimens is not a simple function of the applied stress. In fact the generally increasing displacement of these curves toward the right as stress is decreased indicates that more than the parameters of stress and current crack length must be involved in determining an instantaneous rate of crack propagation. Such an hypothesis has been reported by Weibull (10); our data are currently being analyzed in this regard.

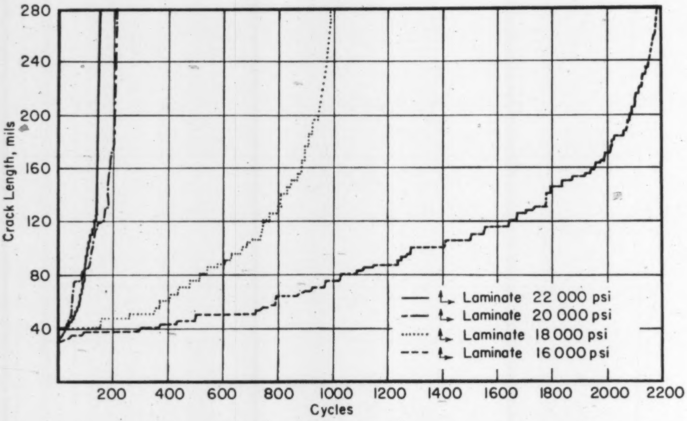


FIG. 7.—Crack Length as a Function of Stress. NY ↑→ Laminate.

Another observation on the fatigue behavior of the laminate structures has to do with the mode of final failure in fatigue. It has been observed that sheet specimens of 1100-H18 aluminum alloy generally fail by the sudden propagation of the crack. Laminates on the other hand begin to tear and fail slowly in tension at the conclusion of a fatigue test. Further, the laminate specimens seem to be able to tolerate a somewhat longer crack (an average of about 0.050 in.) than can the solid sheet specimens.

In this regard, Fig. 8 shows the mode of tensile failure of a laminate of 10 plies of 1100-0 aluminum alloy. Here again the mode of failure is distinctly different than in an equivalent solid sheet. The laminate specimen fails with a much smaller elongation and practically no reduction of area. Failure begins by the formation of a single crack. At constant strain rate (over a wide range) the crack is seen to propagate very slowly until its length is about three-quarters of the specimen width. A second crack then forms on the opposite side of the specimen from the first, and while the first crack remains dormant, the second propagates. Failure eventually occurs in the area between the cracks, or by having the second crack continue to failure. This phenomenon is not yet understood; it is believed that the second crack is caused by a redistribution of stress accompanying the propagation of the first crack and is also affected by a restraint to bending offered by the grips.

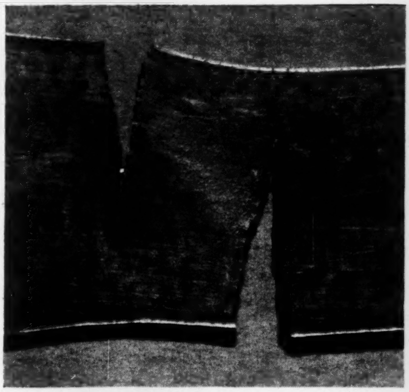


FIG. 8.—Laminated Tension Test Specimen Composed of 10 Plies of 1100-O Aluminum.

This specimen was loaded in tension at a constant strain rate of 0.025 in. per in. per min.

It has been shown that laminated sheet structures have several interesting and potentially valuable properties. They can be made to have the same strength properties as an equivalent solid sheet, and possibly better; they are 7 to 10 per cent less dense than the parent metal foils; and their fatigue behavior indicates that these structures have the facility of retarding crack propagation and possibly crack initiation. Further, they seem to possess an inherent ability to completely halt the propagation of a (normally) catastrophic crack under tensile conditions.

Although such structures as these have been shown to possess several potentially valuable properties, still, the present authors believe that research in this field has barely more than just begun. Before such structures as these can be considered for commercial use they should be further improved and a complete knowledge of their eccentricities gathered. Several approaches to this problem have been indicated here. It is hoped that these and several other ideas will allow the more complete development of these structures.

CONCLUSIONS

As a direct result of this research laminate sheet structures of 1100-H18 aluminum alloy foil bonded with an adhesive layer have been shown to possess the following properties:

1. A modulus of elasticity of 8.5 to 9.4×10^6 psi;
2. A density 7 to 10 per cent less than that of solid aluminum;
3. Static strengths about the same as an equivalent sheet and potentially higher;
4. Fatigue properties which tend to be better than those of a solid sheet at usual

design stresses, on the basis of strength-to-weight ratio; and

5. An inherent crack-retarding ability in fatigue and a crack-arresting ability in tension.

Acknowledgments:

The authors wish to extend their sincere appreciation to Mr. E. Moore, Mr. D. Frank, and Mr. L. Sweeney for their assistance in carrying out the experimental program; to Miss A. Wells, Mr. R. Crowe, and Mr. F. Attinger for their assistance in preparing the data for presentation, to Mrs. Olga Hensler for typing this manuscript, and to Mr. R. Rautio for his technical assistance in designing the modifications of the test equipment.

The interest, advice, and stimulation of Mr. R. Mayerjak (ARL), Mr. E. Callan (ARL), Mr. W. Eberhardt (ALCOA), Mr. L. Mayer (ALCOA), Mr. J. Poynter, Mr. C. A. Davies, Mr. W. Moreland, and Mr. E. J. Hassell, of our laboratory, helped make this research possible.

The technical assistance of the Technical Photographic Laboratory is also gratefully acknowledged.

REFERENCES

(1) W. J. Love, "Structural Changes in Ingot Iron Caused by Plastic and Repeated Stressing," *T. and A. M. Rep. 33*, University of Illinois, Urbana, Ill., Nov. 1952.

(2) G. M. Sinclair and T. J. Dolan, "Use of a Recrystallization Method to Study the Nature of Damage in Fatigue of Metals," *Proceedings* First Nat. Congress of Applied Mechanics, Am. Soc. Mechanical Engrs., p. 647 (1952).

(3) A. J. Fenner, N. B. Owen, and C. E. Phillips, "The Fatigue Crack as a Stress Raiser," *Engineering*, Vol. 171, p. 637 (1951).

(4) W. S. Hyler, E. D. Abraham, and H. J. Grover, "Fatigue Crack Propagation in Severely Notched Bars," *NACA TN 3685*, Nat. Advisory Committee for Aeronautics, June, 1956.

(5) M. S. Hunter and William G. Fricke, Jr., "Cracking of Notch Fatigue Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 643 (1957).

(6) H. A. Lipsitt and G. T. Horne, "The Fatigue Behavior of Decarburized Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 587 (1957).

(7) H. A. Lipsitt, F. W. Forbes, and R. B. Baird, "Crack Propagation in Cold Rolled Aluminum Sheet," see p. 734, this publication.

(8) H. A. Lipsitt, G. E. Dieter, G. T. Horne, and R. F. Mehl, "Study of Effects of Microstructure and Anisotropy on Fatigue of 2024-T4 Aluminum Alloy," *NACA TN 3380*, Nat. Advisory Committee for Aeronautics, March 1955.

(9) D. B. Cross, H. L. Golpen, H. R. Merriman, and M. L. Sheridan, "Development of Adhesive Bonded Metal-to-Metal Laminates," *Engineering Report No. 5870*, Air Material Command, 15 July 1955.

(10) W. Weibull, "Effects of Crack Length and Stress Amplitude on Growth of Fatigue Cracks," *FFA Report 65*, Aeronautical Research Inst. (Sweden), Feb. 1956.

FATIGUE TESTS ON THREE CAST IRONS AT ELEVATED TEMPERATURES

By R. P. Felgar[1]

SYNOPSIS

Direct stress fatigue tests were conducted on three cast irons; Meehanite,[2] nickel-molybdenum, and nodular iron. Pulsating fatigue tests (zero to maximum stress) were performed on all three materials, and completely reversed push-pull tests were conducted on nickel-molybdenum and nodular iron. The specimens were subjected to temperatures from 750 to 1000 F. The staircase method of fatigue testing was used and the data were subjected to statistical analysis.

In pulsating fatigue at 900 F, nodular iron is stronger than Meehanite or nickel-molybdenum iron for 10^7 cycles. Under these same test conditions, Meehanite and nickel-molybdenum iron have essentially the same fatigue strengths.

The tests described in this paper were carried out to determine the fatigue strengths at elevated temperatures for a life of 10^7 cycles of three cast irons, namely, Meehanite, nickel-molybdenum iron, and nodular iron. In service, the material is subjected to a steady stress as well as an alternating stress. For this reason, tests were performed using both pulsating stress cycles and completely reversed stress cycles. To avoid introducing a stress gradient across the normal section of the specimens, axial rather than bending loading was used in all tests. The staircase method of testing, described in reference (**2**)[3] was used throughout.

EXPERIMENTAL PROCEDURE

All specimens, as shown in Fig. 1, were of the same diameter with the exception of those numbered MR1 through MR5; these had test section diameters of 0.400 in. All specimens were tested with the surfaces in the as-machined condition.

All tests were performed on a Sonntag SF 1-U fatigue machine operating at a synchronous speed of 1800 rpm. An axial loading fixture was used which embodied a lever system that produced both a steady and alternating force on the specimen five times as large as the forces exerted at the machine platen. The axial loading fixture was manufactured by the Baldwin-Lima-Hamilton Co., but was modified in the General Engineering Laboratory to decrease the eccentricity of the force applied to the specimen. The modifications consisted of aligning the specimen clamping plates and the addition of flex bars to prevent rotation of the plates to which the specimens were attached. A special set of clamping plates for the specimen was also made.

CONCENTRICITY OF LOAD

The device used to insure concentricity of load is shown in Fig. 2. Each end of the specimen was threaded into a $\frac{1}{2}$-in. thick hardened-steel plate. Steel balls 1 in. in diameter were fitted into the conical seats at each end of the specimen. These balls were also fitted into spherical seats in hardened-steel plates 1 in. thick. The steel plates were pressed against the steel balls by means of four $\frac{1}{2}$-in. diameter, hex head, socket screws. The complete assembly, with the furnace around the specimen, was placed in the device shown in Fig. 2. The specimen rested on two knife-edges, one located in the groove in the specimen to prevent longitudinal motion of the specimen. The eccentricity of the specimen was first checked between the knife-edges; then the screws in the plates were tightened differentially until the maximum run-out of the end plates was 0.001 in. at $2\frac{3}{4}$ in. from the axis of the specimen. With this run-out, the deviation from perpendicularity is 1.25 min. All specimens were aligned in this manner.

---

[1] Mechanics of Materials Engineer, General Engineering Laboratory, General Electric Co., Schenectady, N. Y.

[2] Trade name commercial cast iron.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

Before any test was performed, the alignment fixture and the procedure used to adjust the alignment were checked. This was done by taking strain measurements at points 90 deg apart around the circumference of the test section. A specimen was instrumented with electric resistance strain gages, placed in the loading fixture, and a static load was applied. The strain measurements were taken at room temperature. The maximum bending stress, due to eccentricity, was found to be 4 per cent of the direct stress. This is the maximum bending stress expected in the fatigue tests since the same accuracy of alignment was used for all specimens.

TEMPERATURE CONTROL

A three-winding furnace was used with a Leeds & Northrup temperature indicator and controller. Chromel-alumel thermocouples were used in all tests. The proper voltages for the furnace windings were determined by temperature measurements along the length of the specimen from the fillet at one end of the test section to the fillet at the other end. The temperature did not vary more than ±5 F along the test section and was maintained to within ±5 F of the initial value during the course of the test.

CONTROL OF STEADY AND ALTERNATING FORCE

Since the tests were conducted at elevated temperatures and a mean load was applied, creep occurred. A servo system in the fatigue machine maintained the steady force at its preset value.

The alternating force amplitude remained constant since the compensating weights were selected so that the resonant frequency of the spring-mass system of the fatigue machine exciter was equal to the synchronous speed of the driving motor.

MATERIALS TESTED

The chemical analyses and tensile strengths for the three materials tested as given by the Cooper-Bessemer Corp. are as follows:

	Nodular Iron Heat 3947	Nickel-Molybdenum Iron Heat 1233	Meehanite Heat 1121
Carbon	3.78	3.24	3.27
Silicon	2.45	1.62	1.79
Manganese	0.50	0.71	0.70
Phosphorus	0.066	0.118	0.076
Sulfur	...	0.104	0.093
Nickel	0.78	0.91	1.05
Magnesium	0.105	...	...
Molybdenum	...	0.50	...
Ultimate Tensile Strength, psi	67 000	47 000	43 000

TEST RESULTS

The complete test results are given in this section, followed by a statistical analysis of the data to determine the mean fatigue strength and the standard

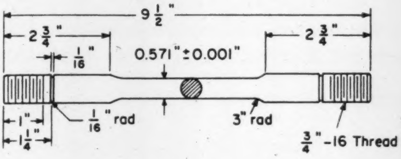


FIG. 1.—Fatigue Specimen.

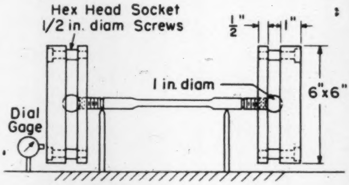


FIG. 2.—Specimen Alignment Device.

deviation. The test data for Meehanite, nickel-molybdenum iron, and nodular iron are given in Tables I, II and III, respectively.

Statistical Analysis:

The mean fatigue limit at 10^7 cycles was computed by two methods: (*a*) Using the statistical equation derived from the staircase procedure as given in reference (2) and (*b*) using the average of the highest stress at which a run-out occurred and the lowest stress at which a failure occurred. The standard deviation was computed from the equation based on the staircase procedure (reference (2)). Since only five tests were used at any given temperature and loading condition, the values given for the standard deviations are very crude estimates of the true standard deviations. The values are given merely as guides to the scatter and should not be used to establish confidence limits or for design purposes. In some cases there was insufficient data to even estimate the standard deviation. The results of the analysis are given in Table IV.

The mean fatigue limits, as computed

TABLE I.—TEST DATA FOR MEEHANITE.
Pulsating Axial Fatigue.

Specimen Number	Temperature, deg Fahr	Maximum Stress, psi	Number of Cycles	Results
CX1...........	750	20 000	10.011×10^6	No failure
CX2...........	750	39 000	1/4	Failed
CX3...........	750	30 000	1×10^4	Failed
CX4...........	750	25 000	2×10^4	Failed
CX5...........	750	20 000	3.7×10^4	Failed
CX6...........	750	15 000	10×10^6	No failure
CX8...........	800	15 000	14.579×10^6	No failure
CX9...........	800	20 000	5.6×10^4	Failed
CX10...........	800	15 000	10×10^6	No failure
CX11...........	800	20 000	3.11×10^5	Failed
CX12...........	800	15 000	3.494×10^6	Failed in threads[a]
CX13...........	900	15 000	12.398×10^6	No failure
CX14...........	900	20 000	1.380×10^6	Failed
CX15...........	900	15 000	15.007×10^6	No failure
CX16...........	900	20 000	10.022×10^6	No failure
CX17...........	900	25 000	4.02×10^5	Failed
CX18...........	1000	15 000	10×10^6	No failure
CX19...........	1000	20 000	7.68×10^5	Failed
CX20...........	1000	15 000	14.893×10^6	No failure
CX21...........	1000	20 000	2.17×10^5	Failed
CX22...........	1000	15 000	14.884×10^6	No failure

[a] Taken as "no failure" at test section.

from the highest stress for run-out and the lowest stress for failure, are plotted against temperature in Fig. 3. The fatigue limits at room temperature, as determined by the Cooper-Bessemer Corp. are also shown for comparison.[4]

TABLE II.—TEST DATA FOR NICKEL-MOLYBDENUM IRON.

Specimen Number	Temperature, deg Fahr	Maximum Stress, psi	Number of Cycles	Results
PULSATING AXIAL FATIGUE				
NX1	900	15 000	10.063×10^6	No failure
NX2	900	20 000	10.118×10^6	No failure
NX3	900	25 000	3.7×10^4	Failed
NX4	900	20 000	15.139×10^6	No failure
NX5	900	25 000	1.14×10^5	Failed
NX6	900	20 000	17.559×10^6	No failure
NX7	1000	20 000	14.750×10^6	No failure
NX8	1000	25 000	3×10^3	Failed
NX9	1000	20 000	17.33×10^6	No failure
NX10	1000	25 000	1.21×10^5	Failed
NX11	1000	20 000	10.361×10^6	No failure
COMPLETELY REVERSED AXIAL FATIGUE				
NX12	900	12 500	15.523×10^6	No failure
NX13	900	15 000	1.47×10^5	Failed
NX14	900	12 500	13.009×10^6	No failure
NX15	900	15 000	12.394×10^6	No failure
NX16	900	17 500	10.363×10^6	No failure

TABLE III.—TEST DATA FOR NODULAR IRON.

Specimen Number	Temperature, deg Fahr	Maximum Stress, psi	Number of Cycles	Result
PULSATING AXIAL FATIGUE				
MX1	900	20 000	15.557×10^6	No failure
MX2	900	25 000	10.001×10^6	No failure
MX3	900	30 000	10.34×10^6	Failed
MX5	900	35 000	5.986×10^6	Failed in threads[a]
MX7	900	38 000	12.601×10^6	No failure
COMPLETELY REVERSED AXIAL FATIGUE				
MR1	900	25 000	2.5×10^4	Failed
MR2	900	22 500	10.105×10^6	No failure
MR3	900	25 000	2.895×10^6	Failed
MR4	900	22 500	11.961×10^6	No failure
MR5	900	25 000	6.210×10^6	Failed

[a] Taken as "no failure" at test section.

DISCUSSION

The matter of greatest interest is the relative fatigue strengths at elevated temperatures of the three cast irons, and the variation in the fatigue strengths with temperature. Figure 3 shows that, for pulsating fatigue, nodular iron is the strongest of the three materials tested at

---

[4] These values were provided by T. E. Eagan of Cooper-Bessemer.

900 F. The nickel-molybdenum iron is only slightly stronger, on the average, than Meehanite. The nodular iron is substantially stronger than the two other materials.

TABLE IV.—STATISTICAL PARAMETERS COMPUTED FROM THE TEST DATA.

Material	Loading Condition	Temperature, deg Fahr	Mean Maximum Stress at 10^7 Cycles		Standard Deviation, psi
			Staircase, psi	Average of Highest and Lowest, psi	
Meehanite	Pulsating	750	20 000	17 500	≈2300
Meehanite	Pulsating	800	17 500	17 500	Insufficient data
Meehanite	Pulsating	900	19 000	20 000	≈2000
Meehanite	Pulsating	1000	17 500	17 500	≈2000
Nickel-molybdenum iron	Pulsating	900	21 200	22 500	≈1800
Nickel-molybdenum iron	Pulsating	1000	22 500	22 500	Insufficient data
Nickel-molybdenum iron	Reversed	900	16 200	16 800	≈2900
Nodular iron	Pulsating	900	34 400	34 400	Insufficient data
Nodular iron	Reversed	900	23 800	23 800	Insufficient data

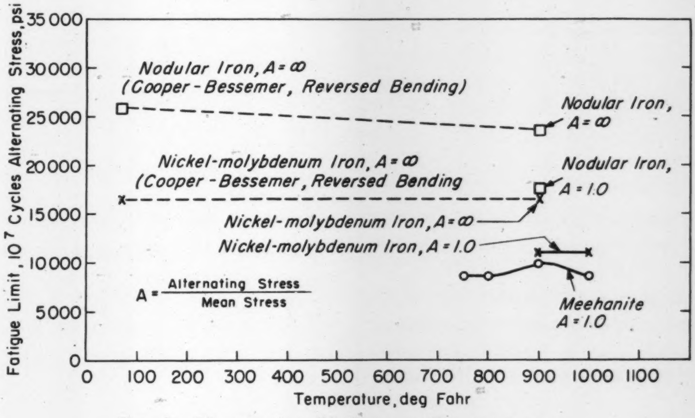


FIG. 3.—Mean Fatigue Limits *versus* Temperature.

For the temperatures used in the experiments, none of the materials exhibited a significant variation in fatigue strength with temperature. Figure 3 shows that, when the Cooper-Bessemer data is used, the reversed stress fatigue strength of nodular iron decreased only 8.5 per cent when the temperature was increased from 70 to 900 F. The reversed stress fatigue limit of the nickel-molybdenum iron is the same at 900 F as at room temperature. The pulsating stress fatigue limit for Meehanite is about 1200 psi greater at 900 F than at either 800 or 1000 F. This is probably not significant since, as shown in Table IV, the standard deviation is about 2000 psi.

A stress range diagram is instructive in assessing the manner in which the fatigue strength is affected by a steady stress. Tests were performed at 900 F with two different ratios of alternating to mean stress (A = 1.0, ∞) for nodular iron and nickel-molybdenum iron. The mean fatigue limits for these test conditions are plotted in Fig. 4. In order to complete the stress-range diagram it is necessary to know the rupture strengths for times equal to the fatigue life of 10^7 cycles divided by the frequency at which the tests were performed. The only data of this kind which were available are discussed below.

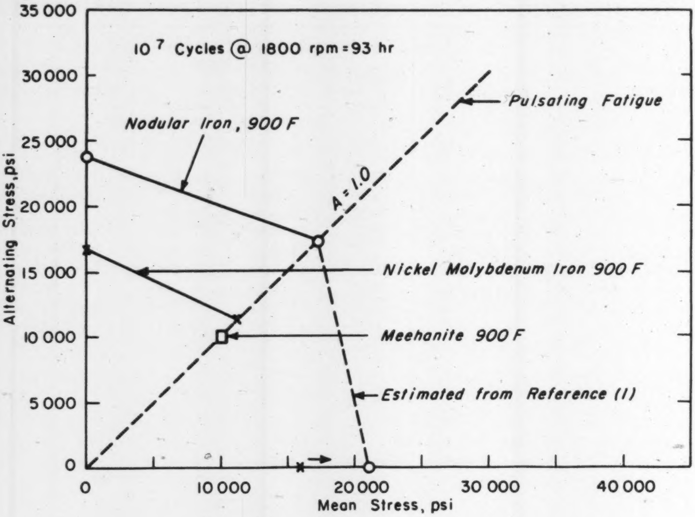


FIG. 4.—Stress Range Diagrams—Axial Fatigue, Temperature 900 F.

In reference (1) the rupture strengths at 800 and 1000 F are given for a nickel-molybdenum and a nodular cast iron having the following chemical compositions:

	Nodular Iron	Nickel-Molybdenum Iron
Carbon	3.56	2.98
Silicon	2.67	1.51
Manganese	0.53	0.57
Phosphorus	0.11	0.11
Sulfur	0.021	0.099
Nickel	0.04	1.56
Magnesium	0.080	<0.01
Molybdenum	<0.01	0.58

These two cast irons are of similar chemical composition to the nodular and nickel-molybdenum irons used in the tests described in this report. The following rupture strengths are given in reference (1) for 93-hr life, corresponding to 10^7 cycles at 1800 rpm.

Material	Temperature, deg Fahr	Rupture Stress, psi
Nodular iron	800	30 000
	1000	12 000
Nickel-molybdenum iron	800	...
	1000	17 000

The rupture strength of nodular iron at 900 F is between 12,000 and 30,000 psi. If the rupture strength varies linearly with temperature, at 900 F it is 21,000 psi. This value is plotted on the mean stress axis in Fig. 4. The rupture strength of the nickel-molybdenum iron at 900 F is probably greater than 17,000 psi, but there are not sufficient data to even estimate its value. In reference (4) rupture strengths are given for a ferritic ductile iron, having a chemical composition almost the same as the nodular iron used in the tests discussed here. The 93-hr rupture strengths at 800 and 1000 F agree with the values given in reference (1).

The nodular iron is distinctly superior in strength at 900 F to the nickel-molybdenum iron for A ratios from 1 to ∞. However, if the 93-hr rupture strength at 900 F of the nickel-molybdenum iron is greater than about 21,000 psi it would be stronger for cases where the alternating stress is small compared to the mean stress.

Figure 4 shows that the stress-range diagrams for nodular iron and nickel-molybdenum iron at 900 F are concave downward. In reference (3), the stress-range diagram for Meehanite, presumably at room temperature, is shown concave upward. The shape of the diagram is determined largely by the point on the mean stress axis. At room temperature the tensile strength is the value plotted on the mean stress axis. However, with increasing temperatures and periods of application of stress, the rupture strength decreases while the fatigue stress is not significantly affected by temperatures over the range used. This effect causes the stress-range diagram to become more sharply concave downward.

CONCLUSIONS

The pulsating fatigue strength at 10^7 cycles of Meehanite is essentially constant from 750 to 1000 F. For nickel-molybdenum iron the pulsating fatigue strength at 10^7 cycles is about the same at 900 and 1000 F. Nodular iron is considerably stronger in pulsating fatigue at 900 F than either Meehanite or nickel-molybdenum iron.

Mean stress has a significant effect on the alternating fatigue strength at 10^7 cycles of both nodular iron and nickel-molybdenum iron at 900 F. The alternating fatigue strengths for both these materials were roughly 30 per cent less for pulsating stress than for completely reversed stress at 900 F.

Acknowledgment:

The cooperation of T. E. Eagan, Chief Research Metallurgist, for the Cooper-Bessemer Corp. is especially appreciated. He was responsible for the preparation of the specimens and also supplied the results of fatigue tests at room temperature on nodular iron and nickel-molybdenum iron. The tests reported here were performed by K. T. Alff of the General Electric Co.

The tests were sponsored by G. M. Wilson of the Locomotive and Car Equipment Department of the General Electric Co.

REFERENCES

(1) "Properties of Cast Iron at Elevated Temperatures," "Progress Report to ASME-ASTM Joint Committee on Effects of Temperature on the Properties of Metals, Southern Research Inst., Birmingham, Ala. (1957).

(2) W. J. Dixon and A. M. Mood, "A Method for Obtaining and Analyzing Sensitivity Data," *Journal*, Am. Statistical Assn., Vol. 43, pp. 109–126 (1948).

(3) T. O. Kuivinen, "Full Scale Fatigue Testing of Compressor Cylinders," Paper presented at the Petroleum Mechanical Engineering Conference of the ASME, Sept. 1952.

(4) C. R. Wilks, N. A. Matthews, and R. W. Kraft, "Elevated Temperature Properties of Ductile Cast Irons," *Transactions*, Am. Soc. for Metals, Vol. 47, pp. 611–631 (1955).

AISI TYPE 304L STAINLESS STEEL WITH IMPROVED STRENGTH*

By R. R. Brady[1]

AISI type 304L stainless steel (0.03 per cent carbon, maximum), which is the low-carbon counterpart of AISI type 304 stainless steel (0.08 per cent carbon, maximum), was developed for use in applications requiring improved resistance to intergranular corrosion. Because of its low carbon content, however, the type 304L steel has lower room- and elevated-temperature strength than does the regular type 304 steel. Therefore, the Subcommittee on Stress Allowances for Ferrous Materials of The American Society of Mechanical Engineers has assigned lower allowable working stresses for type 304L steel than for type 304.[2] This imposes a penalty on the use of type 304L steel in pressure vessels, for example, because the wall thickness of vessels made from type 304L must be greater than that of pressure vessels made from regular type 304.

The U. S. Steel Corp.'s Applied Research Laboratory has investigated the possibility of increasing the room- and elevated-temperature strengths of type 304L steel, without impairing intergranular-corrosion resistance, by the addition of nitrogen. Part of the work concerned with this investigation was conducted at Battelle Memorial Institute and at Armour Research Foundation of the Illinois Institute of Technology under the sponsorship of the U. S. Steel Corp.

Pieces of 3-in. thick plates from commercial electric-furnace heats that had been hot rolled and annealed (1975 F, water quenched) at U. S. Steel Corp.'s Homestead District Works were used in the investigation. As shown in Table I, two of the plates were high-nitrogen type 304L steel (0.10 and 0.13 per cent nitrogen) and one was regular type 304 steel (0.04 per cent nitrogen). The nitrogen content of type 304L steel is normally about the same as that shown in Table I for the regular type 304 steel. The high nitrogen content of the two type 304L steels studied in the present investigation was obtained by adding high-nitrogen ferrochromium to the electric-furnace heats.

Four specimens, $\frac{1}{8}$ in. thick, 1 in. wide, and 2 in. long, were machined from the midthickness of each annealed plate sample and were used for standard Huey corrosion tests. Two of the Huey specimens from each plate were tested in the annealed condition and two were tested in the annealed and sensitized condition. The sensitizing treatment consisted of heating the specimens for 1 hr at 1200 F and air cooling. In the Huey tests at the laboratory, each specimen was tested in boiling 65 per cent nitric acid for five 48-hr periods.

At the laboratory, tension tests were made at room temperature on 0.505-in. diameter longitudinal specimens from the midthickness of the annealed plate samples. At Battelle Memorial Institute, tension tests were made at various temperatures in the range from 200 to 1200 F on 0.254-in. diameter longitudinal specimens also from the midthickness of the plate samples. At Armour Research Foundation, creep-rupture tests were made at 900, 1100, 1300, and 1500 F on standard 0.252-in. diameter longitudinal specimens.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Applied Research Laboratory, U. S. Steel Corp., Monroeville, Pa.

[2] Boiler and Pressure Vessel Code, Am. Soc. Mechanical Engrs., Section VIII, Rules for Construction of Unfired Pressure Vessels, Part UHA, p. 93.

In addition, the laboratory made metallographic studies and Vickers hardness measurements on each steel in the annealed condition and after long-time creep-rupture testing.

The results of the Huey tests, which are presented in Table II, indicate that the intergranular-corrosion resistance of the type 304L steels in the annealed condition was not significantly different from that of the regular type 304 in the same condition. The sensitizing treatment did not significantly affect the corrosion resistance of the low-carbon high-nitrogen type 304L steels but had the usually expected adverse effect on the corrosion resistance of the regular type 304 steel with high carbon and low nitrogen contents. Furthermore, the Huey results for the two high-nitrogen type 304L steels studied in the present investigation were in the ranges previously reported for a series of low-nitrogen commercial type 304L steels, Table III.[3] It is apparent

TABLE I.—COMPOSITION OF THE STEELS INVESTIGATED, PER CENT.
Check Analyses

Steel	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Nickel	Chromium	Nitrogen	Titanium
				HIGH-NITROGEN AISI TYPE 304L					
A..........	0.030	1.47	0.022	0.024	0.64	9.72	19.13	0.10	<0.018
B..........	0.025	1.63	0.026	0.025	0.55	9.87	18.87	0.13	<0.018
				REGULAR AISI TYPE 304					
C..........	0.070	1.62	0.026	0.026	0.79	9.51	18.66	0.04	<0.018

TABLE II.—RESULTS OF HUEY INTERGRANULAR-CORROSION TESTS ON THE HIGH-NITROGEN TYPE 304L STEELS AND ON THE REGULAR TYPE 304 STEEL.

Steel	Condition	Average Penetration, in. per month
	HIGH-NITROGEN—AISI TYPE 304L	
A.....	Annealed	0.0005
	Sensitized 1 hr at 1200 F	0.0011
B.....	Annealed	0.0005
	Sensitized 1 hr at 1200 F	0.0006
	REGULAR AISI TYPE 304	
C.....	Annealed	0.0005
	Sensitized 1 hr at 1200 F	0.0070

NOTE.—The values are the averages of the results obtained on duplicate specimens.

TABLE III.—RESULTS OF HUEY INTERGRANULAR - CORROSION TESTS PREVIOUSLY REPORTED FOR SAMPLES FROM FOUR HEATS OF LOW-NITROGEN TYPE 304L STEEL.

Condition	Range of Average Penetration, in. per month
Annealed..............	0.0005 to 0.0007
Sensitized 2 hr at 1200 F..	0.0005 to 0.0013

---

[3] D. C. Buck, J. J. Heger, F. J. Phillips, and B. R. Queneau, "Corrosion Resistance and Mechanical Properties of Low-Carbon Austenitic Stainless Steels," Symposium on Evaluation Tests for Stainless Steels, Am. Soc. Testing Mats., p. 56 (1949). (Issued as separate technical publication *ASTM STP No. 93.*)

from these results that increasing the nitrogen content of type 304L steel from the normal amount of about 0.04 per cent to about 0.13 per cent does not impair the intergranular-corrosion resistance of the steel as measured by the Huey test.

TABLE IV.—ROOM- AND ELEVATED-TEMPERATURE TENSILE PROPERTIES OF THE HIGH-NITROGEN TYPE 304L STEELS AND OF THE REGULAR TYPE 304 STEELS.

Steel	Test Temperature, deg Fahr	Yield Strength (0.2 per cent Offset), psi	Tensile Strength, psi	Elongation in 1 in., per cent	Reduction of Area, per cent
HIGH-NITROGEN AISI TYPE 304L					
A...	Room	42 700	85 200	59.5	75.0
	200	33 200	73 250	46.5	75.5
	400	25 800	66 000	39.0	73.5
	600	25 400	63 600	36.0	67.5
	800	24 300	62 900	34.0	64.0
	1000	21 100	57 700	35.0	64.0
	1200	18 200	44 900	24.0	36.0
B...	Room	38 400	81 400	60.5	75.5
	200	32 500	72 000	49.0	76.5
	400	25 700	65 000	38.5	72.5
	600	23 500	62 800	35.0	71.0
	800	21 800	60 200	40.5	68.0
	1000	19 900	55 500	34.0	68.5
	1200	19 800	42 400	20.0	29.5
REGULAR AISI TYPE 304					
C...	Room	35 600	80 900	63.5	77.5
	200	33 100	71 400	48.0	75.5
	400	26 300	65 300	38.5	70.5
	600	23 900	64 900	37.0	68.5
	800	21 800	64 300	39.0	69.0
	1000	19 500	58 100	36.0	67.0
	1200	18 300	43 500	31.3	54.5

NOTE.—The values are the averages of results obtained on duplicate specimens.

These Huey tests confirmed the data listed in United States Patent Number 2,602,737 "Corrosion Resisting Steels," issued to W. O. Binder and J. Thompson on July 8, 1952. The data in this patent indicate that the addition of 0.10 per cent nitrogen does not impair the intergranular-corrosion resistance of the balanced normal and low-carbon, 18 per cent chromium, 8 per cent nickel type stainless steels.

The tension test results for the three steels are presented in detail in Table IV, and the yield and tensile strengths are summarized graphically in Fig. 1. Examination of this figure reveals that except for the room-temperature yield and tensile strengths of the 0.10 per cent nitrogen type 304L steel (steel A), which were slightly high, the yield and tensile strengths of the two high-nitrogen type 304L steels were not significantly different from those of the regular type 304

TABLE V.—CREEP-RUPTURE RESULTS FOR THE HIGH-NITROGEN TYPE 304L STEELS AND FOR THE REGULAR TYPE 304 STEEL.

Steel	Test Temperature, deg Fahr	Stress, psi		
		For Rupture in		For Creep Rate of 1 per cent in
		1000 hr	10 000 hr[a]	1000 hr
HIGH-NITROGEN AISI TYPE 304L				
A.....	900	52 000	39 000	51 000
	1100	27 000	21 000	22 000[a]
	1300	11 500	7 900	8 000[a]
	1500	4 600	2 500	2 400[a]
B.....	900	52 000	40 000	50 000
	1100	26 000	19 000	23 000[a]
	1300	11 000	6 700	8 200[a]
	1500	4 700	2 900	2 800[a]
REGULAR AISI TYPE 304				
C.....	900	54 000	47 000	51 000
	1100	27 500	21 000	17 000[a]
	1300	11 500	8 000	6 000[a]
	1500	5 600	3 900	3 400[a]

[a] Determined by extrapolation of data from about 1500 hr.

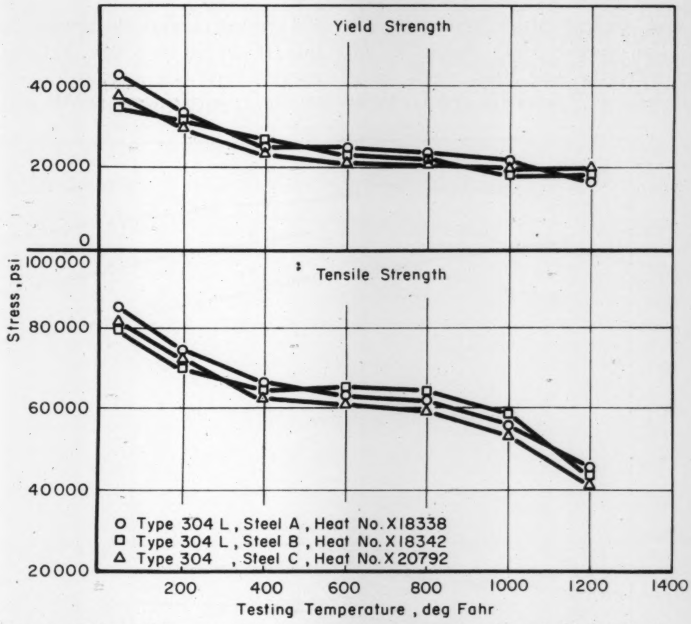


FIG. 1.—Effect of Temperature on the Tensile Properties of the Annealed Steels.

steel over the range of testing temperatures investigated. A comparison of the tensile-ductility results presented in Table IV indicates that the elongation and reduction of area of both the high-nitrogen type 304L steels were the same as those of the regular type 304 steel in the range from room temperature to 1000 F. At 1200 F, however, the elongation and reduction of area of the two type 304L steels were considerably lower than those of the regular type 304 steel. Since the previously tested[3] commercial heats of type 304L steel with normal nitrogen content (0.04 per cent) did not exhibit this deficiency in tensile ductility at 1200 F, it appears that this deficiency in the type 304L steels studied in the present investigation is associated with their high nitrogen contents.

The results of the creep-rupture tests conducted on the three steels are summarized in Table V along with the results from the literature for regular type 304 stainless steel. These results are also presented graphically in Figs. 2 to 5.

The data in Table V indicate that the creep-rupture strengths of the high-nitrogen type 304L steels were higher than those reported in the literature[4] for regular type 304 steel in the range 900 to 1500 F. The data presented in Table V also indicate that except for the 10,000-hr creep-rupture strengths at 900 F and the creep and creep-rupture strengths at 1500 F, the creep-rupture properties of the high-nitrogen type 304L steels were at least equal to those of the regular type 304 steel studied in the present investigation. Although the 10,000-hr creep-rupture strength at 900 F and the 1000- and 10,000-hr creep-rupture strengths at 1500 F of the high-nitrogen type 304L steels were considerably lower than the corresponding strengths of the regular type 304 steel studied in the present investigation, it should be noted that the present regular type 304 steel has much higher strength, particularly at 900 and 1500 F, than that normally exhibited by type 304 steel. This fact can be observed by comparing the data presented in Table V for the type 304 steel studied in the present investigation and the data reported in the literature for type 304 steel.

---

[4] W. F. Simmons and H. C. Cross, "The Elevated-Temperature Properties of Stainless Steels," ASTM-ASME Joint Committee on Effect of Temperature on Properties of Metals, Am. Soc. Testing Mats., (1951). (Issued as separate technical publication *ASTM STP No. 124*.)

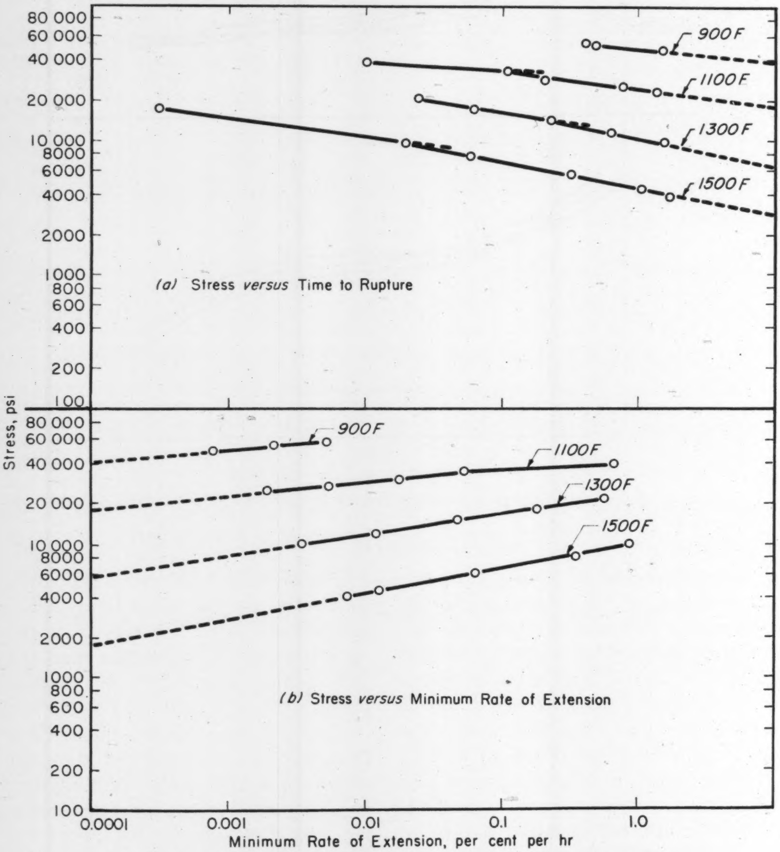


FIG. 2.—Results of Creep Rupture Tests on Type 304L Stainless Steel. Steel A, heat No. X18338; 0.03 per cent carbon, 0.10 per cent nitrogen.

The microstructure of one low-carbon

high-nitrogen type 304L steel (steel A, 0.10 per cent nitrogen) in the annealed condition consisted of austenite, a few undissolved carbides or nitrides, and a small amount of delta ferrite, Fig. 6(*a*), whereas the microstructure of the other low-carbon high-nitrogen type 304L steel (steel B, 0.13 per cent nitrogen) in the annealed condition consisted only of austenite, a few undissolved carbides or nitrides, Fig. 7(*a*). The microstructure of the regular type 304 steel consisted of austenite, undissolved carbides, and a considerable amount of delta ferrite, Fig. 8(*a*). It is generally recognized that nitrogen is a more powerful austenite stabilizer than is carbon, which fact probably accounts for the absence of delta ferrite in the 0.13 per cent nitrogen type 304L steel (steel B), the very small amount in the 0.10 per cent nitrogen type 304L steel (steel A), and the considerable amount

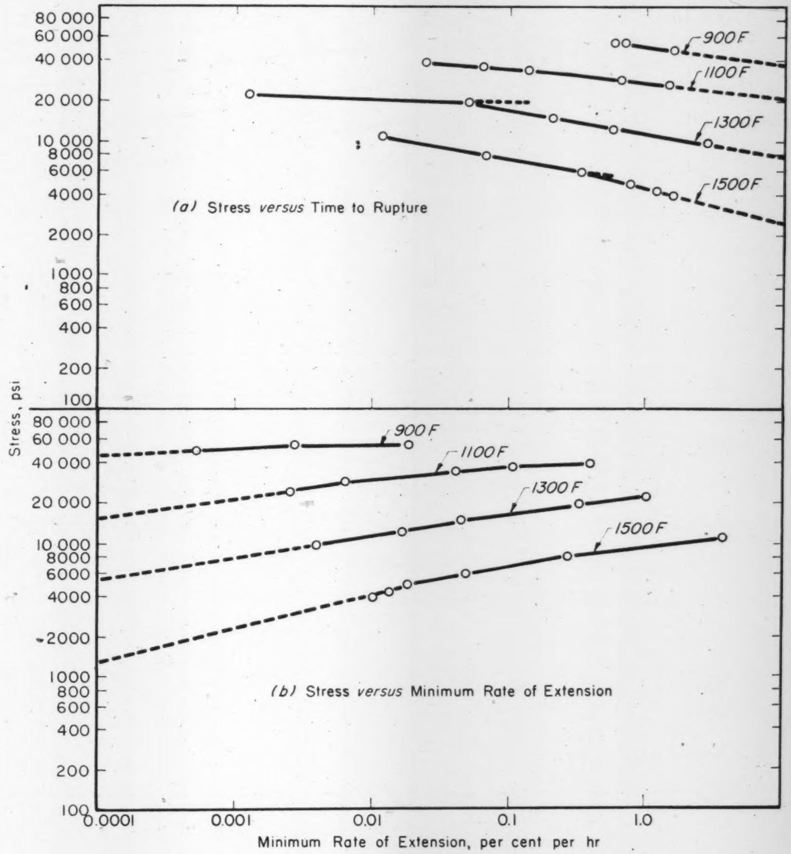


FIG. 3.—Results of Creep Rupture Tests on Type 304L Stainless Steel.
Steel B, heat No. X18342; 0.025 per cent carbon, 0.13 per cent nitrogen.

in the regular type 304 steel (steel C). However, it is entirely possible that a portion of the larger amount of delta ferrite present in the regular type 304 steel (steel C) is also a result of the steel having been heat treated in a somewhat thicker (3-in. thick plate) than normal section.

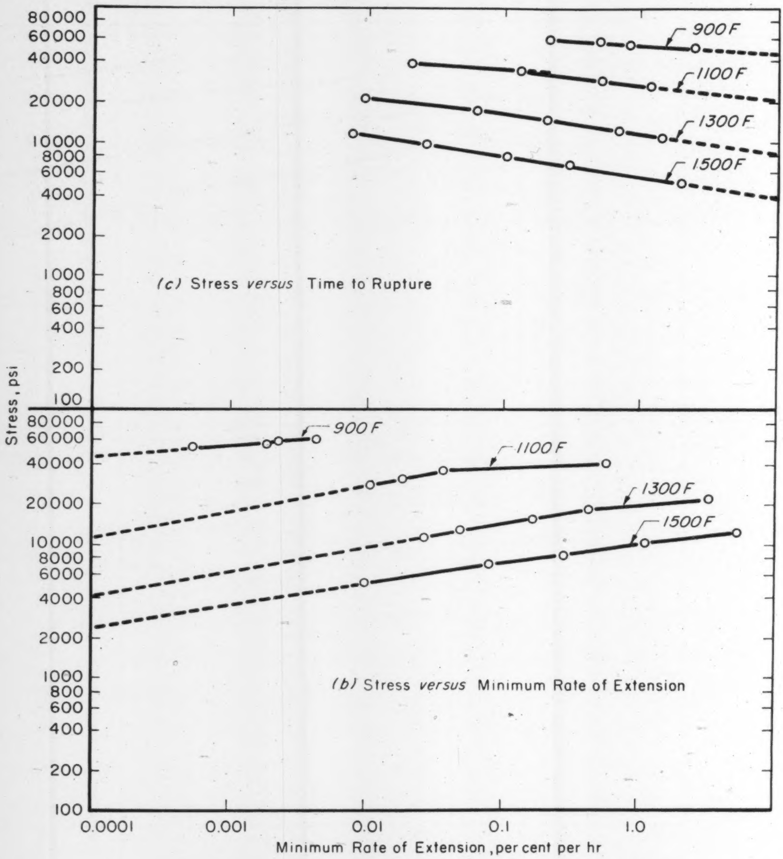
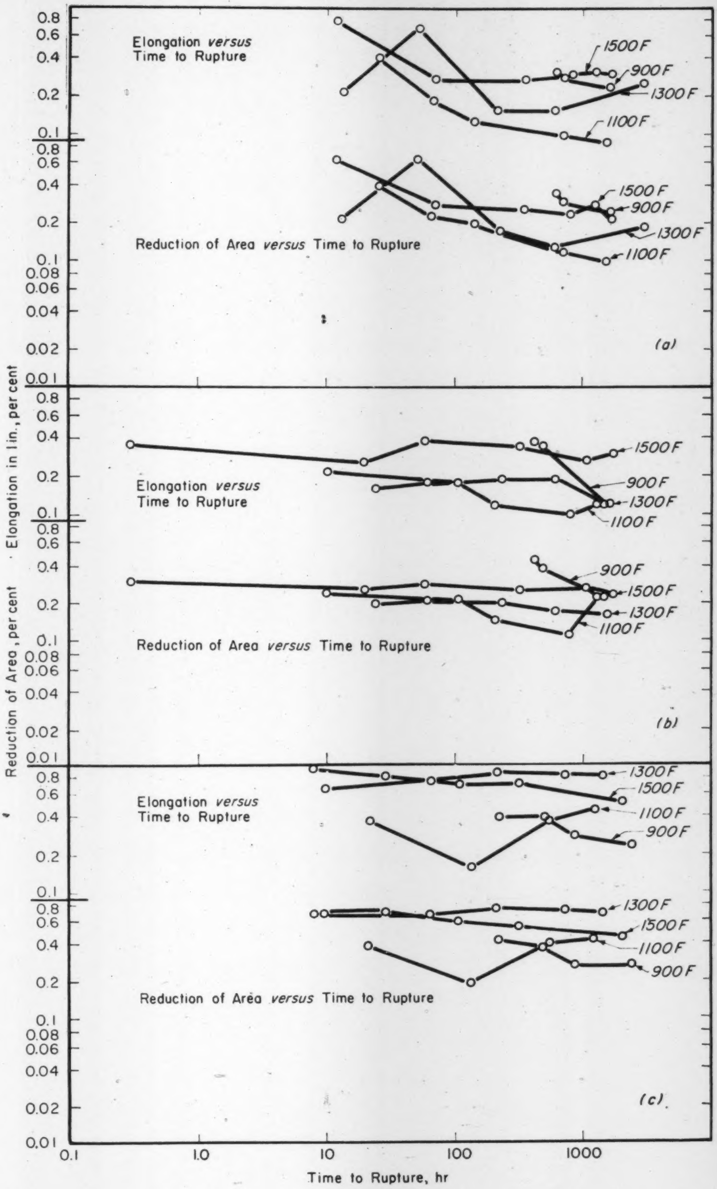


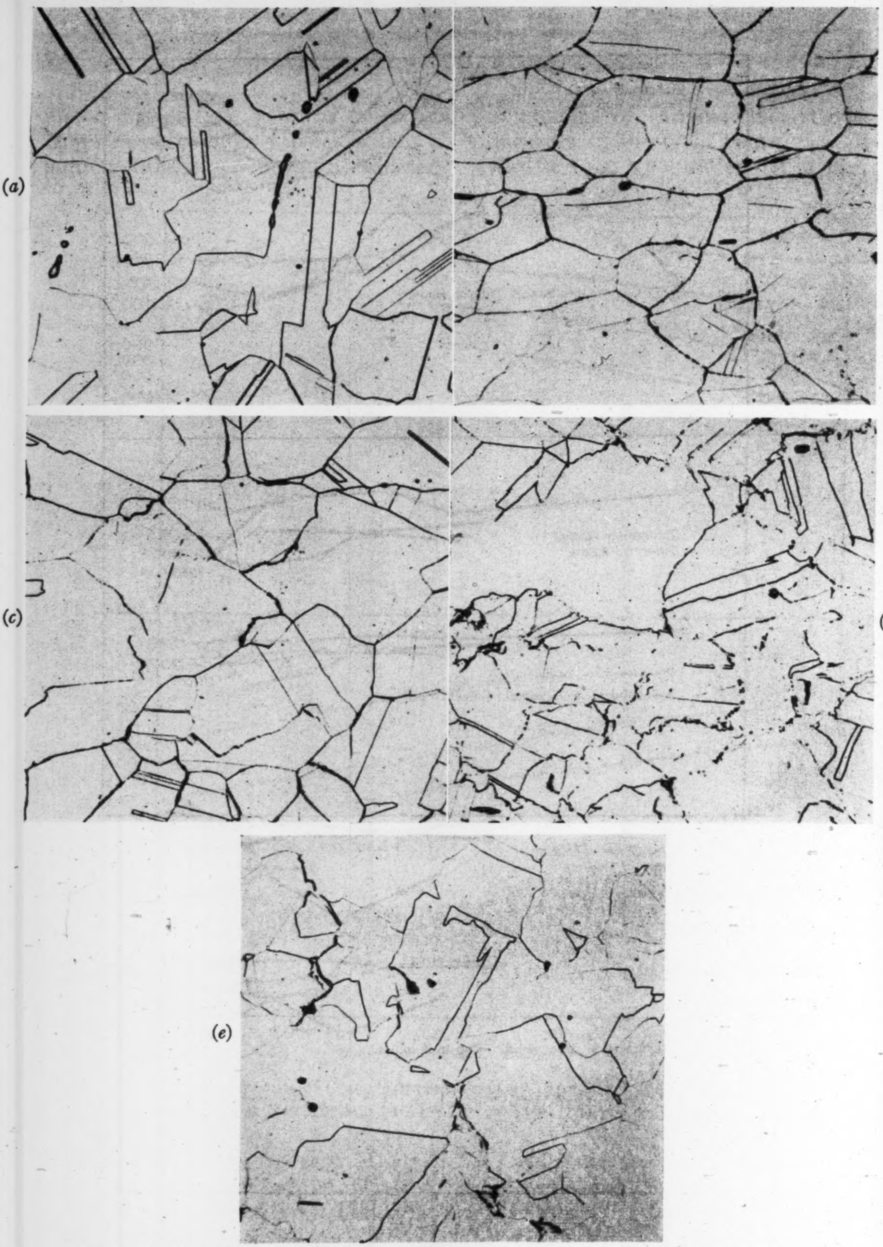
FIG. 4.—Results of Creep Rupture Tests on Type 304 Stainless Steel. Steel C, heat No. X20792; 0.07 per cent carbon, 0.04 per cent nitrogen.

Long-time creep-rupture testing of the high-nitrogen type 304L steels caused precipitation of carbides and nitrides principally at the grain boundaries. As shown in Figs. 6(*b*) to 6(*e*) and in Figs. 7(*b*) to 7(*e*) the amount of this precipitation increased as the testing temperature was increased from 900 to 1500 F. There was considerably less precipitation in the high-nitrogen type 304L steels than in the regular type 304 steel, Fig. 8(*b*) to 8(*e*).



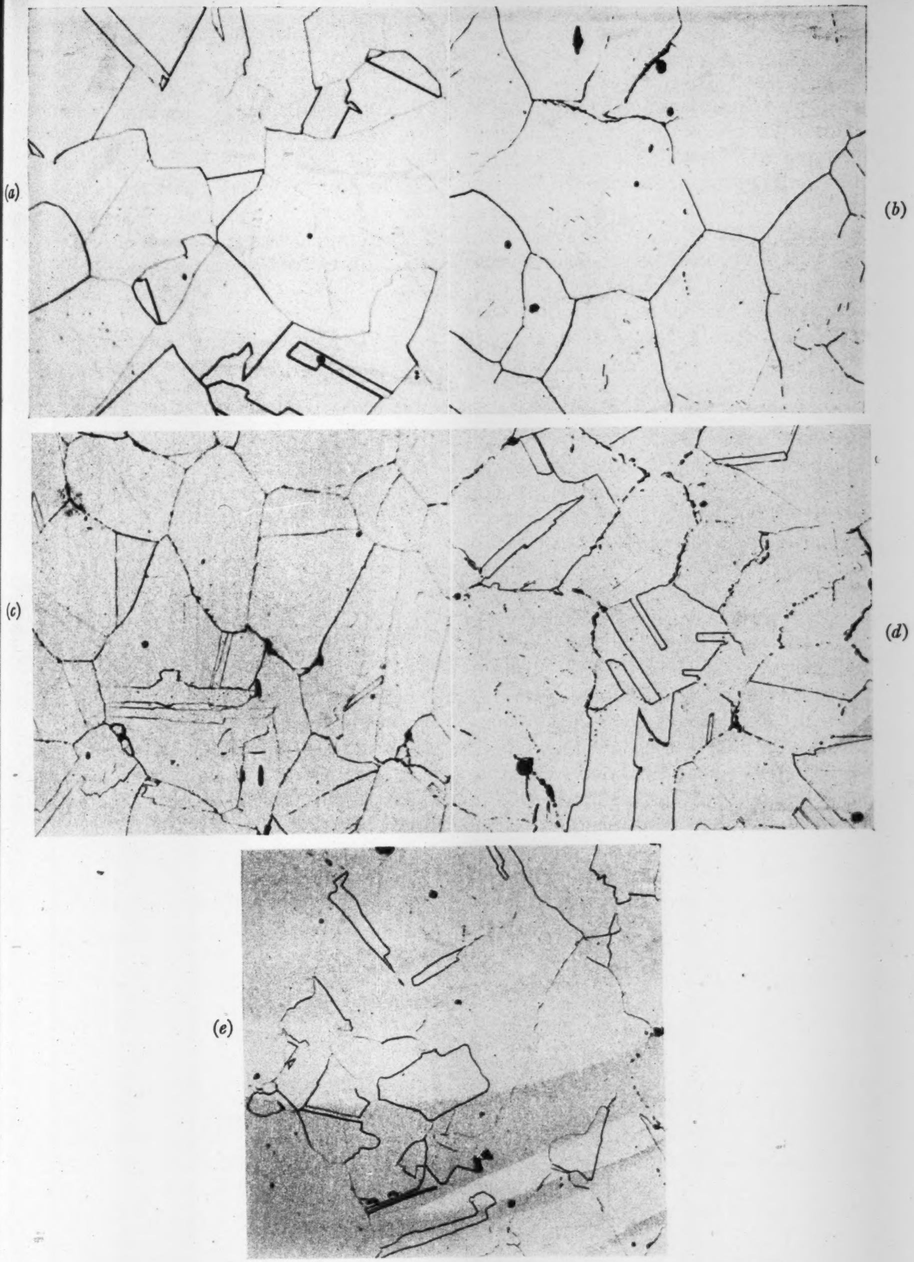
(*a*) Steel A, heat No. X18338; 0.03 per cent carbon, 0.10 per cent nitrogen.
(*b*) Steel B, heat No. X18342; 0.025 per cent carbon, 0.13 per cent nitrogen.
(*c*) Steel C, heat No. X20792; 0.07 per cent carbon, 0.04 per cent nitrogen.

FIG. 5.—Results of Creep Rupture Tests on Type 304L Stainless Steel.



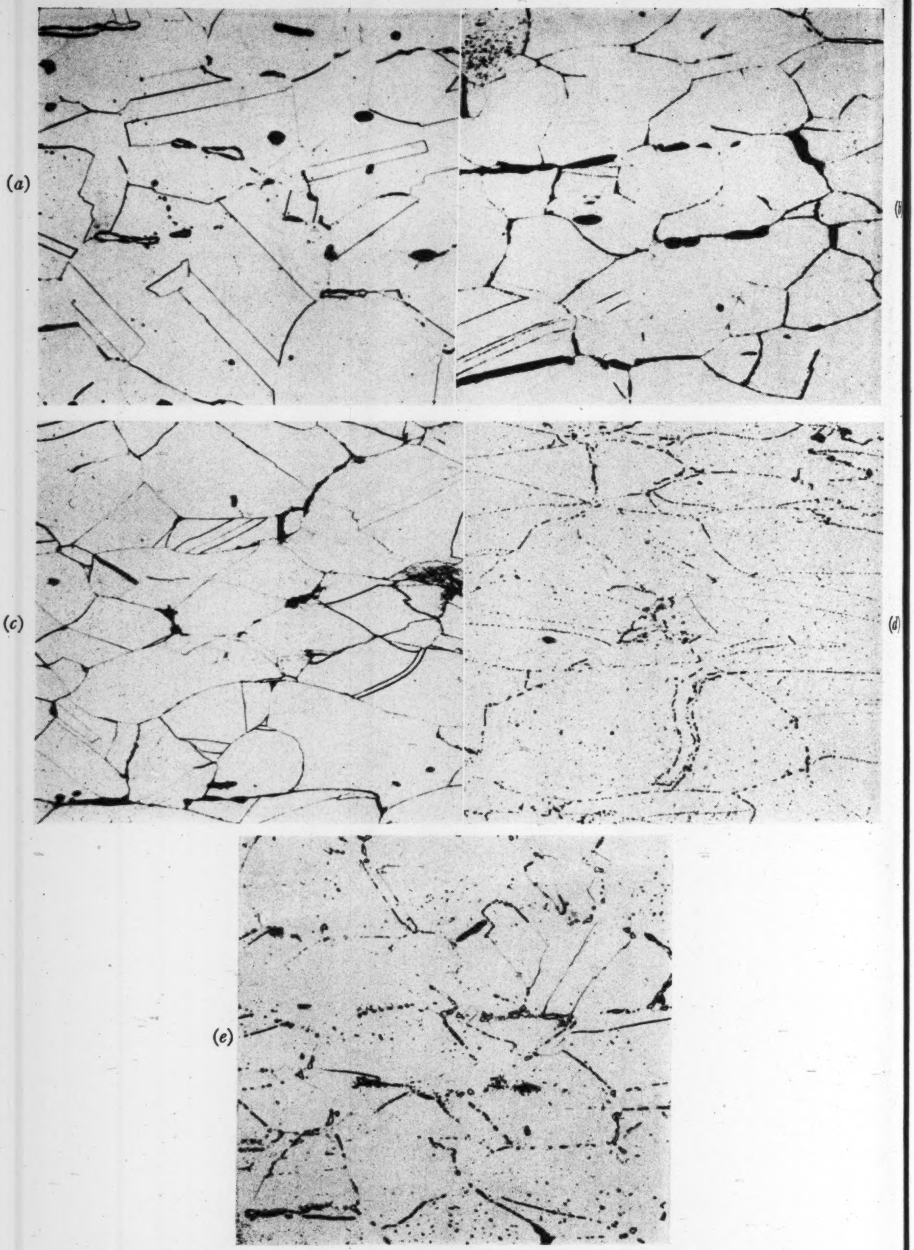
(*a*) Prior to testing. Heat-treated as a 3-in. thick plate by water quenching from 1957 F.
(*b*) After creep-rupture testing for 1523 hr at 900 F. Stress 50,000 psi.
(*c*) After creep-rupture testing for 1463 hr at 1100 F. Stress 25,000 psi.
(*d*) After creep-rupture testing for 2811 hr at 1300 F. Stress 10,000 psi.
(*e*) After creep-rupture testing for 1542 hr at 1500 F. Stress 4000 psi.

FIG. 6.—Microstructure of Type 304L Stainless Steel (steel A, 0.03 per cent carbon, 0.10 per cent nitrogen) After Annealing and After Long-Time Creep-Rupture Testing. Etched in Vilella's reagent.



(*a*) Prior to testing. Heat-treated as a 3-in. thick plate by water quenching from 1975 F.
(*b*) After creep-rupture testing for 1493 hr at 900 F. Stress 50,000 psi.
(*c*) After creep-rupture testing for 1337 hr at 1100 F. Stress 25,000 psi.
(*d*) After creep-rupture testing for 1561 hr at 1300 F. Stress 10,000 psi.
(*e*) After creep-rupture testing for 1700 hr at 1500 F. Stress 4000 psi.

Fig. 7.—Microstructure of Type 304L Stainless Steel (steel B, 0.025 per cent carbon, 0.13 per cent nitrogen) After Annealing and After Long-Time Creep-Rupture Testing. Etched in Vilella's reagent.



(*a*) Prior to testing. Heat-treated as a 3-in. thick plate by water quenching from 1975 F.
(*b*) After creep-rupture testing for 2546 hr at 900 F. Stress 52,000 psi.
(*c*) After creep-rupture testing for 1220 hr at 1100 F. Stress 27,000 psi.
(*d*) After creep-rupture testing for 1428 hr at 1300 F. Stress 11,000 psi.
(*e*) After creep-rupture testing for 2028 hr at 1500 F. Stress 5000 psi.

FIG. 8.—Microstructure of Regular Type 304 Stainless Steel (steel C, 0.07 per cent carbon, 0.04 per cent nitrogen) After Annealing and After Long-Time Creep-Rupture Testing. Etched in Vilella's reagent.

The hardness of all three annealed steels, which was about Rockwell hardness B 85, increased to about Rockwell hardness B 100, after creep-rupture testing at 900 F, and then decreased to values in the range of Rockwell hardness B 70 to 80 as the testing temperature was increased to 1500 F.

TABLE VI.—LITERATURE DATA.

Steel	Test Temperature, deg Fahr	Stress, psi	
		For Rupture in	
		1000 hr	10 000 hr[a]
AISI type 304...	900	49 000	38 000
	1100	25 000	16 800
	1300	10 000	6 100
	1500	3 800	2 350

[a] Determined by extrapolation.

SUMMARY

The present investigation was made to determine whether increasing the nitrogen content of AISI type 304L austenitic stainless steel would provide improved room- and elevated-temperature strength without impairing the steel's good resistance to intergranular corrosion. The results are summarized as follows:

1. In the annealed and in the annealed and sensitized condition, type 304L stainless steel with about 0.10 per cent nitrogen has intergranular-corrosion resistance equivalent to that of normal-nitrogen type 304L steel (0.04 per cent nitrogen) as measured by the Huey test.

2. Except for low tensile ductility at 1200 F, the tensile properties in the range from room temperature to 1200 F of this high-nitrogen type 304L steel are about the same as those of regular type 304 steel (about 0.07 per cent carbon and 0.04 per cent nitrogen).

3. The creep-rupture strengths of the high-nitrogen type 304L steels are superior to those reported in the literature for regular type 304 steel in the range 900 to 1500 F. Except for the 10,000-hr creep-rupture strength at 900 F and the 1000- and 10,000-hr creep-rupture strengths at 1500 F, the creep-rupture strengths of the high-nitrogen type 304L steels are as good as or slightly superior to those of the regular type 304 steel studied in the present investigation. However, the regular type 304 steel studied has much higher strength, particularly at 900 and 1500 F, than that normally exhibited by type 304 steel.

4. In the annealed condition, less delta ferrite is present in the high-nitrogen type 304L steels than in the regular type 304 steel, and the microstructural stability of the high-nitrogen type 304L steels is better than that of the regular type 304 steel.

DISCUSSION

MR. R. A. LULA.[1]—I should like to ask the author if he has any information regarding the weldability of this steel.

MR. R. R. BRADY (*author*).—On a series of tests several years ago on similar steels with this nitrogen content, we did not observe any difficulties in the weldability of the steels.

In addition, we intend to evaluate the hot working characteristics of these nitrogen bearing steels by means of the hot twist test.

---

[1] Chief Research Metallurgist, Allegheny Ludlum Steel Corp., Brackenridge, Pa.

RELATIONSHIPS BETWEEN COMPOSITION AND PROPERTIES OF AUSTENITIC CHROMIUM-MANGANESE-CARBON-NITROGEN STAINLESS STEELS*

By A. Kasak,[1] C. M. Hsiao,[2] and E. J. Dulis[1]

Synopsis

The results of this investigation show that the properties of austenitic Cr-Mn-C-N steels are primarily related to the amounts of carbon and nitrogen in solid solution in the austenite. Therefore, by designing the composition and heat treatment of these steels according to the principles established in this study, a reasonably accurate estimate of the final room- and elevated-temperature properties of the steels can be made.

Because austenitic Cr-Mn-C-N steels have some unique physical and mechanical properties, interest in these steels has been increasing in the past few years. Among the characteristics of these steels that make them attractive are their relatively high room-temperature yield strengths and elevated-temperature strength properties.

For nonmagnetic and elevated-temperature applications as well as for good hot and cold working characteristics of these steels, it is necessary that composition balances be obtained to preclude (1) delta ferrite formation at the solution annealing temperatures and (2) martensite formation during cooling from the solution-annealing, aging, or service temperatures. A recent investigation[3] on Cr-Mn-C-N steels showed that the relationship between the amount of chromium in the steel and the required carbon plus nitrogen to give a completely austenitic structure is as follows:

$$C + N = 0.078\ (Cr - 12.5)$$

To these steels, about 12 to 15 per cent manganese is added to stabilize the austenite against transformation to martensite during cooling to room temperature. Also, in the aforementioned investigation, the relationship between the minimum temperature for complete solution of carbides and the chromium and carbon contents was determined (Fig. 1). Another investigation[4] showed

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Associated with the Central Research Laboratory of Crucible Steel Company of America, Pittsburgh, Pa.

[2] Formerly associated with the Central Research Laboratory of Crucible Steel Company of America, Pittsburgh, Pa.

[3] C. M. Hsiao and E. J. Dulis, "Phase Relationship in Austenitic Chromium-Manganese-Carbon-Nitrogen Stainless Steels," *Transactions*, Am. Soc. Metals, Vol. 50, pp. 773–802 (1958).

[4] C. M. Hsiao and E. J. Dulis, "Effect of Interstitial Carbon Plus Nitrogen and Precipitation Reactions on the Properties of Austenitic Cr-Mn-C-N Steels," *Transactions*, Am. Soc. Metals, Vol. 52, Preprint No. 128.

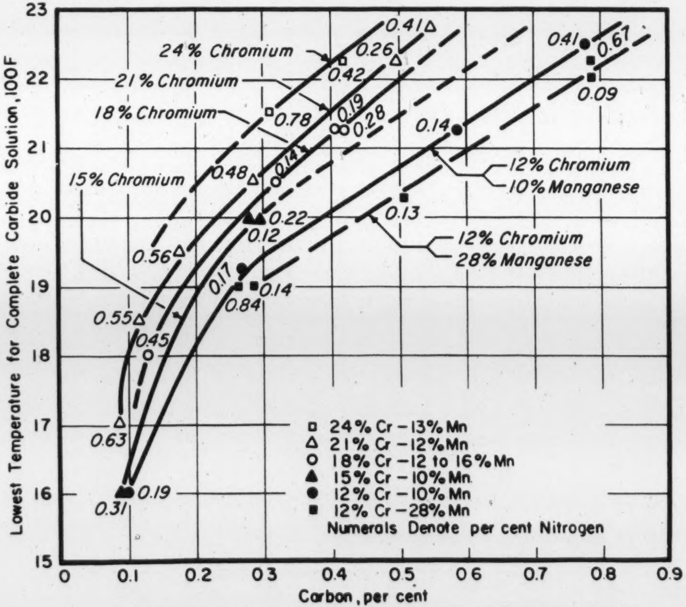


FIG. 1.—Effect of Composition on the Lowest Temperature for Complete Solution of Carbides and Nitrides (footnote 3).

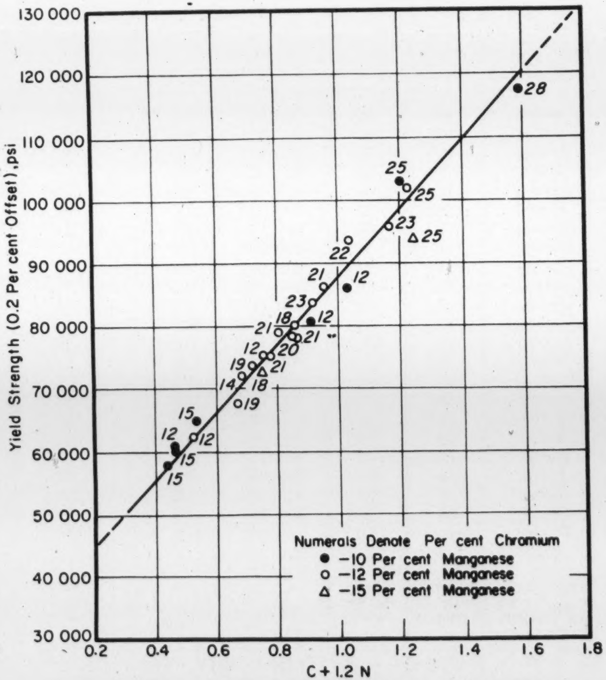


FIG. 2.—Effect of Composition on the 0.2 Per Cent Offset Yield Strength at Room Temperature.

TABLE I.—CHEMICAL COMPOSITIONS OF THE STEELS INVESTIGATED, PER CENT.

Steel	Carbon	Manganese	Chromium	Nitrogen	C + N
No. 1....	0.27	10.47	12.24	0.17	0.44
No. 2....	0.19	12.45	12.25	0.28	0.47
No. 3....	0.78	10.43	12.60	0.21	0.99
No. 4....	0.78	10.31	12.84	0.11	0.89
No. 5....	0.59	11.07	13.39	0.14	0.73
No. 6....	0.32	11.92	13.46	0.30	0.62
No. 7....	0.09	10.60	15.51	0.31	0.40
No. 8....	0.30	10.38	15.62	0.12	0.42
No. 9....	0.28	10.52	15.68	0.22	0.50
No. 10....	0.42	15.75	17.62	0.28	0.70
No. 11....	0.40	12.75	18.42	0.39	0.79
No. 12....	0.30	12.54	18.76	0.35	0.65
No. 13....	0.13	12.98	18.82	0.45	0.58
No. 14....	0.09	12.61	20.15	0.63	0.72
No. 15....	0.29	12.24	21.20	0.41	0.70
No. 16....	0.50	11.52	21.29	0.26	0.76
No. 17....	0.29	12.71	21.35	0.48	0.77
No. 18....	0.76	12.16	21.35	0.17	0.93
No. 19....	0.48	12.26	21.62	0.54	1.02
No. 20....	0.55	11.75	21.87	0.41	0.96
No. 21....	0.82	13.22	22.47	0.29	1.11
No. 22....	0.42	13.11	23.33	0.42	0.84
No. 23....	0.64	12.40	24.42	0.72	1.36
No. 24....	0.31	14.52	24.54	0.78	1.09
No. 25....	0.69	10.60	24.59	0.44	1.13
No. 26....	0.75	12.60	24.84	0.40	1.15
No. 27....	0.81	10.51	27.98	0.66	1.47
No. 28....	0.65	12.46	24.32	0.42	1.07
No. 29....	0.52	12.43	24.02	0.40	0.92
No. 30....	0.39	12.49	23.86	0.68	1.07

TABLE II.—ROOM-TEMPERATURE TENSILE PROPERTIES OF AUSTENITIC CHROMIUM-MANGANESE-CARBON-NITROGEN STEELS.

Steel[a]	Solution-Annealing Temperature, deg Fahr[b]	Rockwell Hardness C Scale	Yield Strength, 0.2 per cent offset, psi	Tensile Strength, psi	Elongation, per cent	Reduction of Area, per cent	Magnetic Response of Fractured Surface
No. 1........	2200	24	64 000	147 000	33	26	Strong
No. 3........	2200	28	88 000	163 000	66	60	None
No. 4........	2200	26	84 000	158 000	66	59	Slight
No. 5........	2200	25	78 000	149 000	68	62	Slight
No. 7........	2100	22	60 000	131 000	36	33	Strong
No. 8........	2100	25	58 000	148 000	52	42	Slight
No. 9........	2100	25	65 000	132 000	60	62	Slight
No. 12........	2100	25	74 000	134 000	59	66	None
No. 13........	2100	23	67 000	120 000	58	64	None
No. 14........	2100	25	78 000	129 000	57	67	None
No. 15........	2100	25	76 000	134 000	62	67	None
No. 16........	2100	31	79 000	148 000	52	54	None
No. 17........	2100	27	78 000	137 000	58	67	None
No. 18........	2100	34	85 000	154 000	33	38	None
No. 20........	2100	35	94 000	160 000	43	48	None
No. 21........	2100	35	94 000	162 000	32	31	None
No. 24........	2100	31	94 000	153 000	60	65	None
No. 25........	2100	38	103 000	170 000	29	33	None
No. 26........	2100	36	100 000	168 000	29	33	None
No. 27........	2200	39	125 000	177 000	27	24	None
No. 28........	2150	34	93 000	161 000	34	33	None
No. 29........	2150	33	88 000	151 000	48	46	None
No. 30........	2150	31	89 000	150 000	52	56	None

[a] The 0.357-in.-diameter, 1.4-in.-gage-length specimens were used on all steels except steels Nos. 28, 29, and 30; on the latter group of steels, specimens with 2-in.-long, 0.40-in.-wide and 0.315-in.-thick rectangular gage sections were used.

[b] All steels were water quenched after solution annealing at the indicated temperatures.

that the the room-temperature yield strengths of stable austenitic Cr-Mn-C-N steels are a function of the parameter (C + 1.2 N), where C and N represent the weight per cent of carbon and nitrogen.

In the present study, the quantitative relationships among (1) composition and room-temperature tensile properties, and (2) composition and 1200 F creep-rupture strength were determined for austenitic Cr-Mn-C-N steels within the following composition ranges:

Carbon	0.09 to 0.82 per cent
Manganese	10.31 to 15.75 per cent
Chromium	12.24 to 27.98 per cent
Nitrogen	0.11 to 0.78 per cent

MATERIALS AND EXPERIMENTAL WORK

The compositions of the Cr-Mn-C-N steels used in this investigation (Table I) are such that all the steels were austenitic after solution annealing and after creep-rupture testing for various times.

Because these steels would undoubtedly be formed and used in the solution-annealed condition, the emphasis of the present study is placed on the properties of these steels in the solution-annealed condition, that is, heated at 2100 to 2200 F and water quenched. Heating of austenitic Cr-Mn-C-N steels in the temperature range of about 1100 to 1800 F results in precipitation reactions that have a marked effect on structure and properties,[4, 5] and a generalization of the properties of these steels after aging is beyond the scope of the present study.

Except for steels Nos. 28, 29, and 30, tension tests were made on either 0.357-in. diameter or 0.505-in. diameter (steels Nos. 10 and 22) specimens, the gage length being four times the diameter. On steels Nos. 28, 29, and 30, the tests were made on 2-in. long by 0.40-in. wide by 0.315-in.-thick (gage dimensions) rectangular specimens. In addition to the tensile properties (tensile strength, 0.2 per cent offset yield strength, elongation, and reduction of area), the magnetic response of the fractured surfaces (to indicate the extent of the austenite-to-martensite transformation) was determined by using either a powerful hand magnet or magne-gage.[6]

The creep-rupture tests were conducted on either 0.357-in. diameter and 1.4-in.-gage length or 0.252-in. diameter and 1.0-in.-gage length specimens.

Conventional testing procedures were used throughout this investigation.

ROOM-TEMPERATURE TENSILE PROPERTIES

The results of tension tests at room temperature (Table II) show that both the tensile and yield strengths increase as the amounts of carbon and nitrogen are increased; the amounts of chromium and manganese, within the ranges studied, have a negligible effect on the tensile properties. The yield strengths are proportional to C + 1.2 N (Fig. 2), and the tensile strengths are proportional to 1.5 C + N (Fig. 3). The deviation of the tensile strength from a linear relationship for the 12 Cr, 10 Mn steels at low parameter values is attributable to the relative instability of the austenitic structure of these steels; the severe plastic deformation during testing caused these steels to transform to martensite. The formation of martensite, particularly in the fracture region of the specimens, has been identified by hardness and magnetic tests as well as by microstructural examinations. This explanation is further substantiated by the observation that no such deviation was

---

[5] C. M. Hsiao and E. J. Dulis, "Precipitation Reactions in Austenitic Chromium-Manganese-Carbon-Nitrogen Stainless Steels," *Transactions*, Am. Soc. Metals, Vol. 49, pp. 655–685 (1957).

[6] The magne-gage is a commercially available instrument for determining the relative magnetic response of specimens.

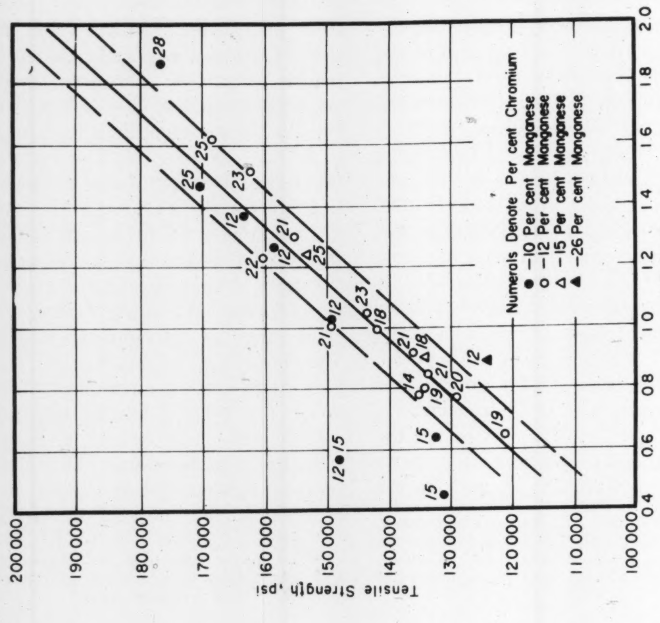


FIG. 3.—Effect of Composition on Tensile Strength at Room Temperature.

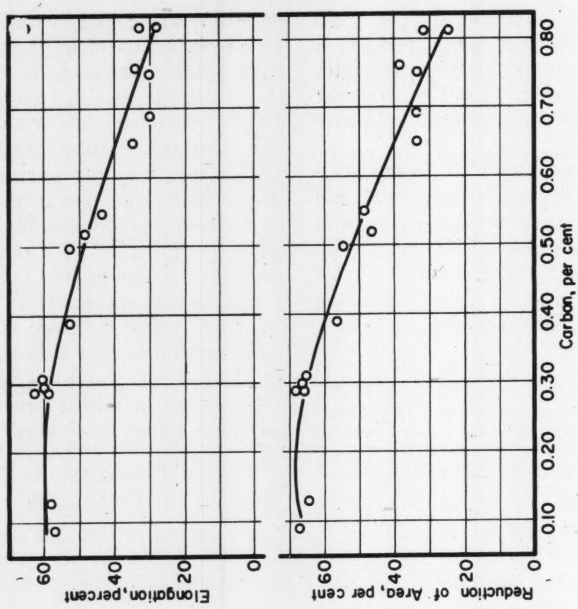
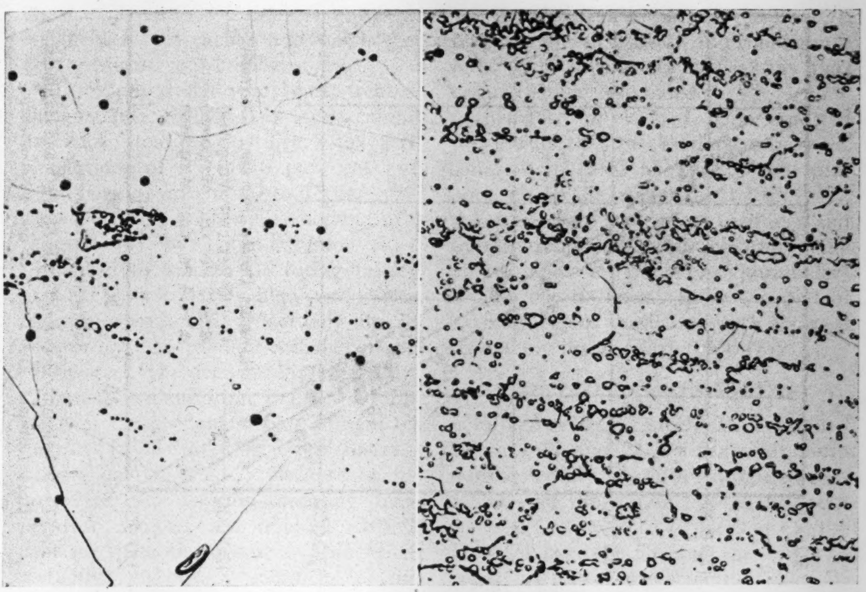


FIG. 4.—Relationship Between Carbon Content and Room-Temperature Ductility of 18 to 27 Per Cent Chromium Austenitic Chromium-Manganese-Carbon-Nitrogen Steels in the Solution-Annealed Condition.



(*a*) Steel No. 30
0.39 per cent carbon, 0.68 per cent nitrogen
Tensile elongation: 52 per cent

(*b*) Steel No. 28
0.65 per cent carbon, 0.42 per cent nitrogen
Tensile elongation: 34 per cent

FIG. 5.—Effect of the Carbon Content on the Microstructure of Austenitic Chromium-Manganese-Carbon-Nitrogen Steels After Solution Annealing at 2100 F and Water-Quenching (× 500).

Etched with 20 per cent HCl in ethyl alcohol.

found for the yield strength relationship of these steels as shown in Fig. 2.

On the basis of the foregoing experimental results, the relationships between the room-temperature strength properties of austenitic Cr-Mn-C-N steels and their carbon and nitrogen contents may be represented by the following linear equations:

$$\text{Tensile strength, psi} = 88{,}400 + 53{,}300\,(1.5\,C + N)$$

$$\text{Yield strength, 0.2 per cent offset, psi} = 34{,}000 + 53{,}500\,(C + 1.2\,N)$$

TABLE III.—EFFECT OF SOLUTION-ANNEALING TEMPERATURE ON TENSILE PROPERTIES AT ROOM TEMPERATURE.[a]

Steel	Solution-Annealing Temperature, deg Fahr[b]	Rockwell Hardness C scale	Yield Strength, 0.2 per cent offset, psi	Tensile Strength, psi	Elongation, per cent	Reduction of Area, per cent	Magnetic Response of Fractured Surface
No. 10........	2100	23	76 000	137 000	58	63	None
	2200	21	73 000	134 000	62	65	None
No. 22........	2100	28	87 000	145 000	47	55	Slight
	2200	25	83 000	143 000	56	59	Slight

[a] Standard 0.505-in.-diameter specimens were used in these tests.
[b] All steels were water-quenched after solution annealing at the indicated temperatures.

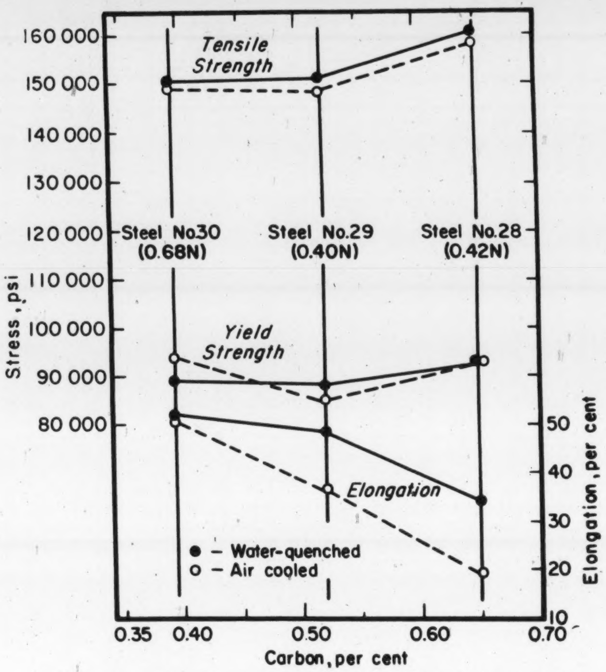


FIG. 6.—Room-Temperature Tensile Properties of Austenitic Chromium-Manganese-Carbon-Nitrogen Steels with Different Carbon and Nitrogen Contents.

The specimens had been annealed at 2150 F for ½ hr. and water-quenched or air cooled.

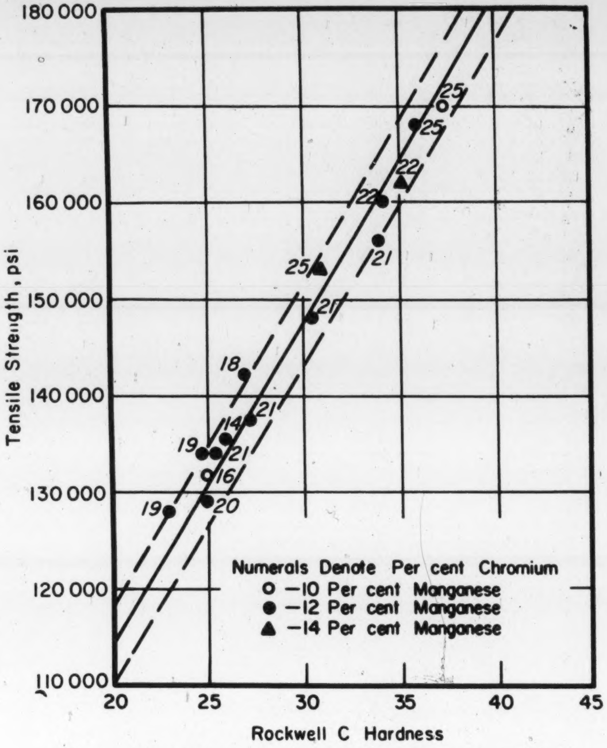


FIG. 7.—Relationship Between Tensile Strength and Hardness. Heat-treatment: 2100 F for ½ hr and water-quench.

In these equations C and N represent weight per cent carbon and weight per cent nitrogen, respectively.

If properly designed and heat treated, the austenitic Cr-Mn-C-N steels exhibit excellent tensile ductility, that is, elongations of 55 to 70 per cent and reductions of area of 50 to 75 per cent. However, the ductility is significantly reduced (1) by strain-induced transformation to martensite during testing and (2) by excessively high carbon and chromium contents, or actually by an excessively high residual carbide content of the steel. The decrease in ductility by carbon is evident from Fig. 4, and the effect of the residual carbide content on ductility is illustrated by the microstructures shown in Fig. 5.[7] Because an increase in the solution-annealing temperature reduces the residual carbide content, some improvement of ductility, virtually without a sacrifice in the strength properties, can be achieved by raising the solution-annealing temperature, for example, from 2100 to 2200 F (Table III).

In addition to the effect of the carbon content, the ductility of stable austenitic Cr-Mn-C-N steels is affected by the rate of cooling from the solution-annealing temperature (Fig. 6). Apparently the decrease in ductility caused by a slow rate of cooling becomes significant at carbon contents in excess of about 0.40 per cent, and the effect is increased as the carbon content is increased. The higher the carbon content of the steel, the greater is its susceptibility to the precipitation of carbides in the form of a continuous grain-boundary network, and the presence of this network has an embrittling effect on the steel. The effect of the cooling rate on ductility is of considerable importance when considering the heat treatment of large sections; if possible, the section size and cooling rate have to be selected in such a way that embrittlement is avoided.

Inasmuch as it had been reported previously[3] that the hardnesses of steels solution annealed at 2100 F were proportional to the parameter (1.5 C + N), the relationship between hardness and tensile strength (found in this study to be proportional to the same parameter) should also be linear. Indeed, a plot of hardness *versus* tensile strength shows a reasonably linear relationship (Fig. 7).

CREEP-RUPTURE PROPERTIES

Fifteen austenitic steels that contained between 12 and 28 per cent chromium were tested in the solution-annealed condition, and the results for the 12 to 18 per cent Cr, the 18 to 21 per cent Cr, the 22 to 24 per cent Cr, and the 25 to 28 per cent Cr steels are listed in Tables IV to VII, respectively. The 100-hr rupture strengths at 1200, 1300, 1400, and 1500 F were interpolated from the master rupture curves for these steels and are listed in Table VIII.

It is evident from Table VIII that all experimental steels are stronger than type 316 stainless steel and that some steels of the series (steels Nos. 23, 27, and 19) are stronger than the 16 Cr, 25 Ni, 6 Mo alloy. The differences among the steels with regard to the 100-hr rupture strength are significant at 1200 F (for example, 55,000 psi for steel No. 23 and 35,000 psi for steel No. 9), but the differences become negligible as the temperature is increased to 1400 and to 1500 F. In general, the results given in Table VIII indicate that the rupture strength is related to the amount of carbon and nitrogen dissolved in the austenite rather than to the total amount of carbon and nitrogen in the steel. This conclusion is substantiated by the observation that the rupture life at 1200 F of steel No. 27 under a stress of 45,000 psi increased from 36 to 319 hr when the solution-annealing temperature was increased from 2000 to 2200 F.

---

[7] Note that the C + N content of the steels shown in Fig. 5 is the same, namely 1.07 per cent.

To establish the association among the structural changes that occurred during test, the type of fracture, and the creep-rupture properties of Cr-Mn-C-N steels, the fractured ends of all the tested creep-rupture specimens were examined microscopically. The following generalizations were established from the results of the microstructural studies:

1. Except for the specimens that ruptured in a few hours, all specimens exhibited equiaxed grains, transverse intergranular voids, and typically intergranular fractures.
2. Externally applied stresses favored the general precipitation within the grains and retarded the grain-boundary reaction.
3. Externally applied stress changed the morphology of the lamellar precipitate; the lamellae tended to grow in the direction perpendicular to the direction of applied stress.

COMPOSITIONAL DESIGN OF STABLE AUSTENITIC CHROMIUM-MANGANESE-CARBON-NITROGEN STEELS

Based on the preceding data, the compositions of stable austenitic Cr-Mn-C-N steels may be designed by the following procedure:

1. Determine the amount, per cent, of chromium required for the indicated service conditions (for example, temperature and allowable oxidation).

2. From the amount, per cent, of chromium required and the following equation, calculate the combined amount, per cent, of carbon plus nitrogen needed:

$$C + N = 0.078\ (Cr - 12.5)$$

3. Proportion the amounts of carbon and nitrogen with regard to the following considerations:

(*a*) the amount of nitrogen that could be put into the steel during melting by conventional methods,

(*b*) the solution-annealing temperature, and

(*c*) the desired room-temperature strength and ductility.

4. Stabilize the austenite against transformation to martensite by adding about 12 per cent manganese when the chromium content is over 15 per cent and 12 to 18 per cent manganese when the chromium content is less than 15 per cent.

5. Estimate the room- and elevated-temperature strengths. These strengths can be controlled by changing the amounts of carbon and nitrogen.

6. Increase the chromium and/or manganese content (and thereby the nitrogen solubility) if the amount of nitrogen desired is beyond the limit that could be put into the steel during melting.

A nomograph (Fig. 8) can be used to facilitate the procedures to be followed (*a*) for the compositional design of stable austenitic Cr-Mn-C-N steels and (*b*) for the estimation of room- and elevated-temperature properties of these steels. The procedure to be followed and some typical examples in using the nomograph are as follows:

Compositional Design of Stable Austenitic Cr-Mn-C-N Steels:

Chromium versus Carbon and Nitrogen.—Any straight line which passes through scales (I), (II), (III), and (IV) would give the minimum amount of carbon plus nitrogen required for austenitic steels at different amounts of chromium.

EXAMPLE A: 21 per cent chromium requires a minimum amount of 0.67 per cent (C + N), which would be apportioned to different amounts of carbon and nitrogen

TABLE IV.—CREEP AND CREEP-RUPTURE PROPERTIES OF 12 PER CENT TO 18 PER CENT CHROMIUM AUSTENITIC STEELS.

Steel	Carbon, per cent	Manganese, per cent	Chromium, per cent	Nitrogen, per cent	*HT*[a]	Test Conditions		Results			
								Rupture			Minimum Creep Rate, per cent per hr
						Temperature, deg Fahr	Stress, psi	Life, hr	Elongation, per cent	Reduction of Area, per cent	
No. 2.....	0.19	12	12	0.28	A	1200	45 000	10	16	20	0.28
					A	1350	30 000	14	12	18	...
					A	1450	20 000	14	8	25	...
No. 6.....	0.32	12	14	0.30	A	1200	45 000	31	8	14	...
					A	1350	25 000	85	11	22	...
					A	1500	13 500	71	24	30	...
No. 9.....	0.28	10	16	0.22	A	1200	45 000	5.5	16	19	1.0
					A	1250	35 000	20	11	15	0.24
					A	1300	25 000	83	11	20	0.065
					A	1350	25 000	40	13	18	0.17
					A	1450	15 000	55	14	16	0.13
No. 10.....	0.42	16	18	0.28	B	1200	60 000	0.5	15	29	...
					B	1200	45 000	15	7	12	...
					A	1200	45 000	23	7	11	...
					A	1200	40 000	55	7	6	0.055
					B	1200	30 000	>1310[b]	3[b]	2[b]	0.0027
					B	1400	45 000	0.1	24	34	...
					B	1500	20 000	7.5	13	16	...
					A	1500	20 000	9	13	25	...

[a] *HT* = Heat Treatment: A—Solution annealed at 2100 F for ½ hr and water quenched.
B—Solution annealed at 2200 F for ½ hr and water quenched.

[b] Discontinued without rupture.

TABLE V.—CREEP AND CREEP-RUPTURE PROPERTIES OF 18 PER CENT TO 21 PER CENT CHROMIUM AUSTENITIC STEELS.

Steel	Carbon, per cent	Manganese, per cent	Chromium, per cent	Nitrogen, per cent	*HT*[a]	Test Conditions		Results			
						Temperature, deg Fahr	Stress, 1000 psi	Rupture			Minimum Creep Rate, per cent per hr
								Life, hr	Elongation, per cent	Reduction of Area, per cent	
No. 11.......	0.40	12	18	0.39	A	1200	65 000	2.5	19	23	...
					A	1200	45 000	87	8	8	0.032
					A	1300	30 000	121	11	14	...
					A	1400	20 000	103	11	14	...
					B	1500	13 500	69	17	18	0.10
No. 12.......	0.30	13	19	0.35	A	1200	45 000	13	12	12	0.31
					A	1250	35 000	32	8	12	0.12
					A	1300	25 000	155	8	8	0.037
					A	1350	25 000	40	9	11	0.17
					A	1450	15 000	99	12	14	0.13
No. 14.......	0.09	13	20	0.63	A	1200	45 000	65	16	18	0.11
					A	1250	35 000	75	11	12	0.090
					A	1350	25 000	45	12	16	0.13
No. 17.......	0.29	13	21	0.48	A	1200	45 000	29	9	13	0.11
					A	1250	35 000	105	8	10	0.052
					A	1300	25 000	236	9	12	0.022
					A	1350	25 000	94	9	11	0.059
					A	1450	15 000	84	12	14	0.065
No. 19.......	0.48	12	21	0.54	A	1200	50 000	157	7	9	...
					A	1300	35 000	160	11	15	0.043
					A	1450	20 000	30	16	16	...

[a] *HT* = Heat Treatment: A—Solution annealed at 2100 F for ½ hr and water quenched.
B—Solution annealed at 2200 F for ½ hr and water quenched.

TABLE VI.—CREEP AND CREEP-RUPTURE PROPERTIES OF 22 PER CENT TO 24 PER CENT CHROMIUM AUSTENITIC STEELS.

Steel	Carbon, per cent	Manganese, per cent	Chromium, per cent	Nitrogen, per cent	*HT*[a]	Test Conditions		Results			
						Temperature, deg Fahr	Stress, psi	Rupture			Minimum Creep Rate, per cent per hr
								Life, hr	Elongation, per cent	Reduction of Area, per cent	
No. 21.......	0.82	13	22	0.29	A	1200	45 000	22	14	20	0.46
					A	1250	35 000	50	21	24	0.20
					A	1300	25 000	90	20	30	0.32
					A	1350	25 000	51	19	27	0.20
					A	1450	15 000	83	22	20	...
No. 22.......	0.42	13	23	0.42	B	1200	60 000	2	16	20	...
					B	1200	45 000	56	6	10	...
					A	1200	45 000	57	8	8	...
					B	1300	35 000	35	11	15	0.16
					A	1300	35 000	29	14	18	0.26
					B	1350	25 000	146	10	14	0.054
No. 23.......	0.64	12	24	0.72	B	1200	60 000	36	7	7	0.079
					B	1200	45 000	782	3	4	...
					B	1350	35 000	20	11	12	...
					B	1450	20 000	16	22	20	...
					B	1450	20 000	14	7	16	...
					B	1600	10 000	16	22	17	...

[a] *HT* = Heat Treatment: A—Solution annealed at 2100 F for ½ hr and water quenched.
B—Solution annealed at 2200 F for ½ hr and water quenched.

TABLE VII.—CREEP AND CREEP-RUPTURE PROPERTIES OF 25 PER CENT TO 28 PER CENT CHROMIUM AUSTENITIC STEELS.

Steel	Carbon, per cent	Manganese, per cent	Chromium, per cent	Nitrogen, per cent	*HT*[a]	Test Conditions		Results			
						Temperature, deg Fahr	Stress, psi	Rupture			Minimum Creep Rate, per cent per hr
								Life, hr	Elongation, per cent	Reduction of Area, per cent	
No. 24.......	0.31	15	25	0.78	A	1200	45 000	78	10	12	...
					A	1250	35 000	47	14	13	...
					A	1350	25 000	17	15	16	0.19
					B	1450	15 000	20	27	21	0.35
					B	1600	8 000	23	29	21	0.49
No. 26.......	0.75	13	25	0.40	A	1200	45 000	64	13	12	0.13
					B	1200	45 000	80	7	7	0.05
					A	1250	35 000	103	20	24	0.11
					A	1300	25 000	124	17	24	0.09
					A	1350	25 000	30	28	37	0.40
					A	1450	15 000	67	13	16	...
					B	1600	10 000	27	15	18	0.23
No. 27.......	0.81	10	28	0.66	B	1200	60 000	42	14	17	0.21
					B	1200	45 000	319	10	11	0.020
					B	1300	35 000	73	13	18	0.11
					B	1400	25 000	20	17	25	0.56
					C	1200	60 000	13	21	20	0.89
					C	1250	35 000	49	21	26	0.14
					C	1350	25 000	15	37	32	0.69
					D	1200	45 000	36	15	34	0.18

[a] *HT* = Heat Treatment: A—Solution annealed at 2100 F for ½ hr and water quenched.
B—Solution annealed at 2200 F for ½ hr and water quenched.
C—Solution annealed at 2200 F for ½ hr and air cooled.
D—Solution annealed at 2000 F for ½ hr and water quenched.

TABLE VIII.—100-HR CREEP-RUPTURE STRENGTHS OF SIXTEEN AUSTENITIC CHROMIUM-MANGANESE-CARBON-NITROGEN STEELS.

Steel[a]	Carbon, per cent	Manganese, per cent	Chromium, per cent	Nitrogen, per cent	C + N	(C + N)[g]	100-hr Rupture Strength, psi at			
							1200 F	1300 F	1400 F	1500 F
No. 23[b]	0.64	12	24	0.72	1.36	1.10	55 000	35 000	18 000	11 000
No. 27[b]	0.81	10	28	0.66	1.47	0.91	54 000	33 000	18 000	...
No. 27[c]	0.81	10	28	0.66	1.47	0.91	42 000	24 000	13 000	...
No. 19	0.48	12	21	0.54	1.03	0.90	52 000	37 000	21 000	12 000
No. 11	0.40	12	18	0.39	0.79	0.77	44 000	31 000	20 000	13 000
No. 26	0.75	13	25	0.40	1.15	0.73	43 000	27 000	17 000	11 000
No. 24	0.31	15	25	0.78	1.09	1.01	43 000	23 000	14 000	9 000
No. 22[d]	0.42	13	23	0.42	0.84	0.80	43 000	31 000	22 000	...
No. 14	0.09	10	20	0.63	0.72	0.72	42 000	28 000	18 000	...
No. 17	0.29	13	21	0.48	0.77	0.77	40 000	28 000	18 000	11 000
No. 6	0.32	12	14	0.30	0.62	0.62	40 000	29 000	19 000	13 000
No. 10[b]	0.42	16	18	0.28	0.70	0.70	38 000	30 000	20 000	...
No. 21	0.82	13	22	0.29	1.11	0.66	38 000	26 000	18 000	12 000
No. 2	0.19	12	12	0.28	0.47	0.47	37 000	28 000	18 000	12 000
No. 12	0.30	10	19	0.35	0.65	0.65	36 000	26 000	18 000	12 000
No. 9	0.28	13	16	0.22	0.50	0.50	35 000	25 000	17 000	11 000
Type 316[e]	0.05	...	18	...	...	...	32 000	21 000	14 000	10 000
Type 16-25-6[f]	...	...	16	...	...	...	45 000	30 000	20 000	14 000

[a] All steels except those indicated were solution annealed at 2100 F and water quenched.
[b] Solution annealed at 2200 F and water quenched.
[c] Solution annealed at 2200 F and air cooled.
[d] Steel No. 22 contained small amounts of ferrite.
[e] Type 316 also contains 12 per cent nitrogen and 2 per cent molybdenum.
[f] Type 16-25-6 also contains 25 per cent nitrogen and 6 per cent molybdenum.
[g] Amount of (C + N) in solution as calculated from Fig. 1.

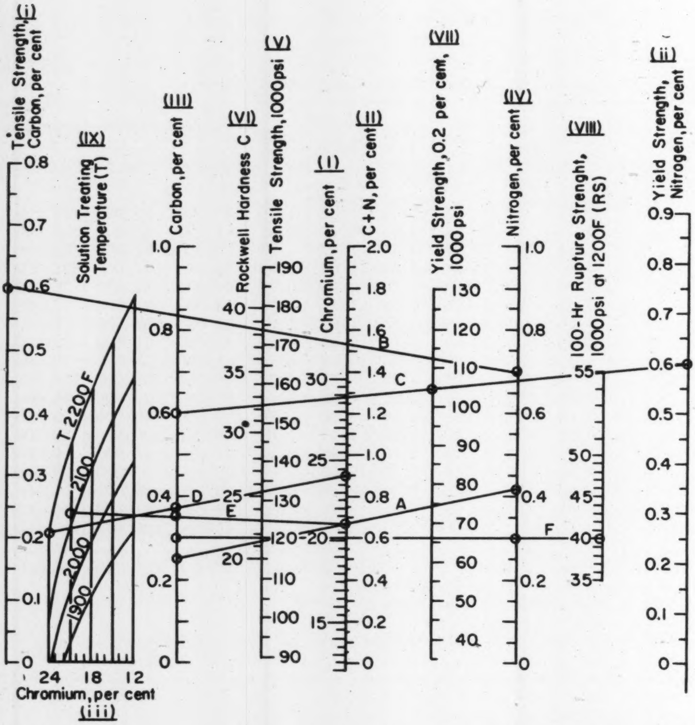


FIG. 8.—Nomograph for Compositional Design of Austenitic Chromium-Manganese-Carbon-Nitrogen Steel and for Estimation of Their Room- and Elevated-Temperature Strengths.

The examples given in this nomograph are identified by the same symbols (A-F) as in the text.

(for example, 0.25 per cent carbon and 0.42 per cent nitrogen).

Manganese.—12 per cent manganese is required for a chromium content over 15 per cent, or 12 to 18 per cent manganese for a chromium content below 15 per cent.

Estimation of Room-Temperature Tensile Properties (for Stable Austenitic Steels Only):

General Remarks.—The effects of chromium and manganese are negligible; steels are in 2100 to 2200 F solution-annealed condition.

Tensile Strength and Hardness.—Any straight line which passes through scales (i), (IV), (V), and (VI) would give the corresponding values of carbon in per cent, nitrogen in per cent, tensile strength in 1000 psi, and hardness in Rockwell hardness C.

EXAMPLE B: For 0.6 per cent carbon and 0.7 per cent nitrogen, the tensile strength and hardness are 173,000 psi and Rockwell hardness C 38 respectively.

0.2 Per Cent Offset Yield Strength.—Any straight line which passes through scales (ii), (III), and (VII) would give the corresponding values of nitrogen in per cent, carbon in per cent, and 0.2 per cent offset yield strength in 1000 psi.

EXAMPLE C: For 0.6 per cent carbon and 0.6 per cent nitrogen, the 0.2 per cent offset yield strength is 105,000 psi.

Elongation and Reduction of Area.—If practically all carbides are in solution (see next section), both the elongation and reduction of area are more than 50 per cent.

Solution-Annealing Treatment:

Nitrides in the amount corresponding up to 0.7 per cent nitrogen in the steel could be dissolved by solution annealing at 1900 F, and the effect of manganese (in the range of 12 to 18 per cent) on the solubility of carbides is small.

EXAMPLE D: How much carbon in a 0.45 carbon-12 manganese-24 chromium-0.45 nitrogen steel could be taken into solution by a 2200 F solution-annealing treatment? Locate the intersection of scale (iii) at 24 per cent chromium and of scale (IX) at 2200 F; connect this point with the chromium content (24 per cent) on scale (I); the intercept of this line on scale (III) gives the per cent carbon in solution (0.37 per cent).

EXAMPLE E: Determine the lowest solution-annealing temperature for complete solution of carbides in a 0.35 carbon-12 manganese-21 chromium-0.35 nitrogen steel. Connect the corresponding points of per cent chromium and per cent carbon on scales (I) and (III), respectively, read *T* on the intersection point with scale (iii) at 21 per cent chromium—2100 to 2150 F.

100-hr Rupture Strength at 1200 F (for a Ratio of Carbon to Nitrogen of About 1:1):

The effects of chromium and manganese are small.

Any straight line perpendicular to scales (II) and (VIII) would give the corresponding values of (C + N) and 100-hr rupture strength at 1200 F.

EXAMPLE F: For 0.3 per cent carbon and 0.3 per cent nitrogen, 40,000 psi is given.

Acknowledgments:

The authors are grateful to the management of Crucible Steel Company of America and to the Materials Laboratory, Wright Air Development Center, for granting permission to publish this paper. This work was largely done under Wright Air Development Center Contract No. AF33(616)-3318. Also, they are pleased to acknowledge their gratitude to J. H. Moll, J. L. Boelky, and other members of the staff who aided in various phases of the experimental work.

DISCUSSION

Messrs. K. G. Brickner and R. R. Brady[1] (*presented in written form*).—Although the paper did not indicate the size of the heats studied nor the type of furnace in which the heats were melted, we have been informed by the authors that the heats were small laboratory induction-furnace heats. It, therefore, would be of interest to determine whether the equations presented for the determination of the room-temperature yield strength [Y. S. = 34,000 + 53,500 (C + 1.2 N)] and for the determination of the room-temperature tensile strength [T. S. = 88,400 + 53,300 (1.5 C + N)] would hold for annealed material processed from commercial size electric-furnace heats. In this connection, the discussors used the aforementioned equations to calculate the room-temperature yield and tensile strengths for sheet and plate material processed from two commercial electric-furnace heats of USS Tenelon stainless steel (0.10 per cent carbon, 14.5 per cent manganese, 17.0 per cent chromium, 0.40 per cent nitrogen). In addition, to determine the usefulness of the equations in calculating these properties for Cr-Mn-C-N steels containing additional alloying elements, the equations were also used to calculate the yield and tensile strengths of material from commercial electric-furnace heats of AISI types 201 (0.15 per cent carbon maximum, 6.5 per cent manganese, 17.0 per cent chromium, 4.5 per cent nickel, 0.25 per cent nitrogen maximum), AISI type 202 (0.15 per cent carbon maximum, 8.75 per cent manganese, 18.0 per cent chromium, 5.0 per cent nickel, 0.25 per cent nitrogen maximum), and USS 17-5 MnV (0.10 per cent carbon, 15.0 per cent manganese, 5.0 per cent nickel, 17.0 per cent chromium, 2.0 per cent molybdenum, 0.75 per cent vanadium, 0.35 per cent nitrogen) stainless steel.

The results of the calculations indicate that reasonable estimates of the actual yield strength and tensile strength of a Cr-Mn-C-N steel (such as Tenelon stainless steel) commercially processed to sheet and plate form can be obtained with these equations. For example, the calculated yield strength of one of the heats of Tenelon stainless steel was 68,800 psi, whereas the actual yield strength of this steel, depending on the annealing time used, was 68,200 to 74,400 psi. Also, the calculated tensile strength was 120,400 psi and the actual tensile strength was in the range 122,700 to 126,100 psi. Similar results were obtained on the other heat of Tenelon stainless.

A similar agreement was observed between the calculated and actual room-temperature yield and tensile strengths of the type 201 and type 202 stainless steels. Thus, the author's equations are valid for the type 200 series stainless steels, and it appears that nickel in amounts up to 5 per cent does not significantly affect the room-temperature yield nor tensile strength of Cr-Mn-C-N steels.

However, for USS 17-5 MnV steel, a Cr-Mn-C-N steel containing nickel, molybdenum, and vanadium as additional alloy additions, the agreement between the calculated and actual yield and tensile strengths is not nearly as good. For example, the calculated yield and tensile strengths of a heat of USS 17-5 MnV steel were 62,900 and 116,600 psi, respectively, whereas the actual yield and tensile strengths of this heat were 85,400 and 134,000 psi, respectively. From these results, one may conclude that, as would be expected, when these Cr-Mn-C-N steels contain a combination of several additional alloying elements, such as nickel, molybdenum, and vanadium, the equations are no longer valid. Perhaps the authors have already considered this fact and are planning additional work to determine a suitable equation for the more highly alloyed Cr-Mn-C-N steels.

---

[1] U. S. Steel Corp., Applied Research Laboratory, Monroeville, Pa.

MR. A. KASAK (*author*).—The heats made for this investigation were 30-lb induction-melted heats.

As the discussers, we also have tested samples from commercial-size heats of different austenitic Cr-Mn-C-N steels and found that the relations presented in the paper are reasonably well applicable.

Of course, the inapplicability of our equations to the Cr-Mn-C-N steels containing appreciable amounts of other additional alloying elements is to be expected. In our experience, additional elements may lead to considerable strengthening in the annealed condition and alter drastically the aging characteristics of these steels; the strong carbide formers (such as vanadium, molybdenum, etc.) have a particularly significant effect. However, we have not studied the effects of the additional alloying elements quantitatively and have no such work planned.

PROPERTY RELATIONSHIPS OF SOME CAST AND FORGED CHROMIUM-MANGANESE-NICKEL-NITROGEN STEELS CONTAINING 18 PER CENT CHROMIUM*

By K. Haefner,[1] A. F. Lahr,[1] W. L. Meinhart,[1] and J. J. Kanter[1]

Synopsis

Some properties of steels containing 18 per cent chromium, 0 to 18 per cent manganese, 0 to 8 per cent nickel, 0 to 0.5 per cent nitrogen, and approximately 0.08 per cent carbon were determined. Austenitic compositional ranges permitting the casting of gas-free ingots were found to be a function of a proper balance of the elements manganese, nickel, and nitrogen.

Nickel exhibited a marked influence on the corrosion-resistance of these steels. Manganese showed little effect and nitrogen indicated a dual behavior.

Nitrogen strengthened the steels and lowered their ductility slightly. Manganese had little influence, and nickel showed two different effects on mechanical properties when alloyed into nitrogen-free and nitrogen-containing steel compositions.

Pronounced embrittlement occurred in low nickel-containing steel compositions in the temperature range of 1000 to 1600 F. It was found to be due to sigma-phase formation from ferrite and phenomena preceding this change in steel compositions containing more than 5 per cent ferrite. No embrittlement was detected in 1000 hr at 800 F in steels containing up to 15 per cent ferrite.

In highly austenitic steels the embrittlement consisted of grain boundary and lamellar precipitation containing Cr_4C, Cr_2N, or both. After long-time annealing at 1800 F, nitrogen-containing steel compositions indicated a localized or section sensitive embrittlement.

The austenite-forming ability of the element manganese in chromium steels has been known for some time. Investigations of steels of this type by Schafmeister and Ergang (**3**)[2] and others (**1,2,5**) showed manganese to be less effective than nickel in its ability to form and stabilize austenite. Ferrite-free structures were attained only in steels containing less than 15 per cent chromium. Austenite containing more than 13 per cent chromium was found to be of a semistable nature when exposed to long-time annealing at temperatures of 900 to 1500 F.

Recently, much attention has been given to the austenitic steels containing more than 15 per cent chromium, employing austenite-forming elements such as manganese, nickel, nitrogen, and combinations thereof. For example, Krainer (**4**) claims stable austenitic structures for steels containing 18 per cent chromium, 5 to 8 per cent manganese, and

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Crane Co., Engineering Laboratories, Research Division, Chicago, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

6 per cent nickel. Carney (8) describes fully austenitic steels within the compositional range of 15 to 20 per cent chromium, 12 to 20 per cent manganese, 0 to 3 per cent nickel, and 0.25 to 0.45 per cent nitrogen; he shows that increasing amounts of nitrogen extend the austenite to austenite plus ferrite phase boundary to higher temperatures. Franks et al (6) presents structural relationships, mechanical and corrosion studies of steels containing 12 to 18 per cent chromium, 1 to 22 per cent manganese, 0 to 14 per cent nickel, and 0 to 0.18 per cent nitrogen.

A review of published data has indicated the need for investigation of the properties of steels containing 0 to 18 per cent manganese, 0 to 8 per cent nickel, 0 to 0.5 per cent nitrogen, and approximately 0.08 per cent carbon at the 18 per cent chromium level. Hence, phase relationships, nitrogen solid solubility (liquid-solid), forgeability, castability, and corrosion resistance of these alloys were studied. Embrittlement tests were conducted and properties in both the cast and forged condition were evaluated.

EXPERIMENTAL PROCEDURE

A series of 150 heats was made, comprising compositional ranges shown in Table I. Chemical analyses of the alloys conformed closely with the nominal. Examples are cited in Table I.

TABLE I.—COMPARISON OF SOME NOMINAL AND CHEMICAL ANALYSES, PER CENT

COMPOSITIONAL RANGE:

Carbon, 0.08; silicon, 0.3 to 0.5; chromium, 18; nickel, 0 to 8; manganese, 0 to 18; nitrogen, 0 to 0.5

Nominal Analyses						Chemical Analyses									
Carbon	Silicon	Chromium	Nickel	Manganese	Nitrogen	Carbon	Silicon	Chromium	Nickel	Manganese	Nitrogen	Magnesium	Molybdenum	Sulfur	Phosphorus
0.08	0.4	18	0.4	17	0	0.09	0.42	17.54	0.47	16.88	0.05	0.01	0.01	0.015	0.009
0.08	0.4	18	0.4	16	0.24	0.07	0.42	18.02	0.24	15.75	0.27	0.02	0.01	0.015	0.009
0.08	0.4	18	0.4	17.5	0.50	0.07	0.33	18.51	0.39	16.72	0.45	0.01	0.01	0.013	0.009
0.08	0.4	18	2	3	0	0.06	0.40	18.06	2.24	2.80	0.03	0.01	0.01	0.008	0.008
0.08	0.4	18	2	11	0.25	0.09	0.42	18.01	2.00	11.20	0.26	0.01	0.01	0.008	0.010
0.08	0.4	18	2	14	0.35	0.10	0.37	18.46	2.12	14.20	0.32	0.01	0.01	0.011	0.008
0.08	0.4	18	4	8	0	0.06	0.42	17.96	4.09	8.40	0.03	0.01	0.01	0.011	0.008
0.08	0.4	18	4	11	0.15	0.07	0.44	18.04	4.09	10.55	0.18	0.01	0.01	0.010	0.008
0.08	0.4	18	4	14	0.45	0.09	0.49	18.26	3.85	13.65	0.41	0.01	0.01	0.011	0.008
0.08	0.4	18	6	11	0	0.09	0.44	17.74	6.21	11.80	0.04	0.01	0.01	0.009	0.009
0.08	0.4	18	6	11	0.15	0.06	0.42	17.47	6.21	12.00	0.14	0.01	0.01	0.007	0.008
0.08	0.4	18	6	11	0.35	0.08	0.40	17.94	6.36	11.15	0.32	0.01	0.01	0.007	0.008
0.08	0.4	18	8	1	0.25	0.07	0.40	18.04	8.12	0.97	0.22	0.02	0.01	0.011	0.008
0.08	0.4	18	8	14	0.50	0.07	0.37	18.20	7.71	14.48	0.45	0.01	0.01	0.008	0.008
0.08	0.4	18	8	17	0.35	0.07	0.40	17.70	7.85	16.56	0.34	0.01	0.01	0.010	0.008

Preparation of Alloys and Samples:

The alloys for this investigation were made from ingot iron, electrolytic manganese, electrolytic nickel, low-carbon ferrochrome, and nitrogen-bearing ferrochrome. Induction furnaces of 12- and 200-lb capacity were used in the preparation of alloys. Although manganese was present in large amounts, additional deoxidation was performed with ferrosilicon (75 per cent silicon) and nickel magnesium (80 per cent nickel, 20 per cent magnesium). Twelve - pound, 3 - inch round; and 30-lb, 3 by 3-in. hot-topped ingots were poured respectively. Each ingot was inspected visually for gassiness and, subsequently, radiographed. A 1-in. thick slice was cut from the 12-lb ingots for cast property evaluation and the remainder forged into 1-in. round bars for room temperature mechanical, corrosion, and delta-ferrite determinations. The 30-lb ingots were forged into $1\frac{1}{4}$-in. square bars for embrittlement studies.

Cast and forged $\frac{1}{2}$-in. thick specimens for structural investigations were heat-treated at temperatures of 1700, 2000, and 2300 F for 2 hr and water-quenched. Forged bars for room temperature mechanical and corrosion testing were heat-treated for 2 hr at 2000 F, followed by water quenching. Charpy impact blanks for the embrittlement studies were cut from the $1\frac{1}{4}$-in. square forged bars, solutionized at 2000 F for 2 hr and water-quenched. The embrittlement investigation was made at temperatures of 800, 1000, 1200, 1400, 1600, and 1800 F for time periods up to 1000 hr. Specimens were removed periodically to follow the progress of the embrittlement process.

Testing Methods:

1. Austenite and ferrite relationships were determined from metallographic structures by lineal analyses and by response of specimens to a magnetic torsion balance.

2. The upper limit of nitrogen solubility upon solidification was indicated visually and radiographically by presence or absence of gas-hole porosity.

3. Mechanical properties at room temperature were obtained from tension, Charpy impact, and Vickers hardness tests, using standard 0.505-in. diameter tensile bars and keyhole notch impact specimens.

4. Corrosion properties were determined using Huey and Strauss tests as per ASTM Tentative Recommended Practice (A 262).[3] Test specimens $\frac{3}{4}$ by $\frac{3}{4}$-in. diameter and $\frac{1}{4}$ by $\frac{1}{2}$ by $3\frac{1}{2}$-in. were employed respectively.

5. Embrittlement effects were evaluated by keyhole notch Charpy impact tests and hardness surveys at room temperature. Structural changes were followed metallographically and precipitation products analyzed by X-ray diffraction of ferro-chloride extracts.

Results and Discussion

Nitrogen Solid Solubility:

Considerable nitrogen may be retained in solution in a given molten alloy composition. During solidification some of it may be liberated as a gas due to a decrease in solubility with the change of state and, therefore, render an ingot gassy. The term, nitrogen solid solubility, as used in the following discussion describes the capacity of a given alloy to hold nitrogen in solution without gas evolution upon solidification. The effect of manganese and nickel on nitrogen solid solubility for 10-lb ingots is graphically presented in Fig. 1. From this it is apparent that manganese-nickel-nitrogen relationships exist permitting the casting of compositions free from nitrogen gas porosity.

The nitrogen solid solubility remains constant with manganese increasing to some minimum content. Thereafter, the ability of the alloy to keep nitrogen in solution increases with increase in manganese. With each increasing nickel content, lower minimum manganese levels are required. It is evident that the elements manganese and nickel used synergistically increase the nitrogen solid solubility.

---

[3] Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 - 55 T), 1958 Book of ASTM Standards, Part 3, p. 288.

Carney (8), in his investigation of nickel-free austenitic stainless steels, found that there was a minimum manganese level for a given chromium content and nitrogen content below which nitrogen evolution occurred during solidification. The results of the present investigation, however, indicate that the nitrogen solubility relationships for the nickel-containing chromium-manganese-nitrogen steels at 18 per cent chromium are more complex.

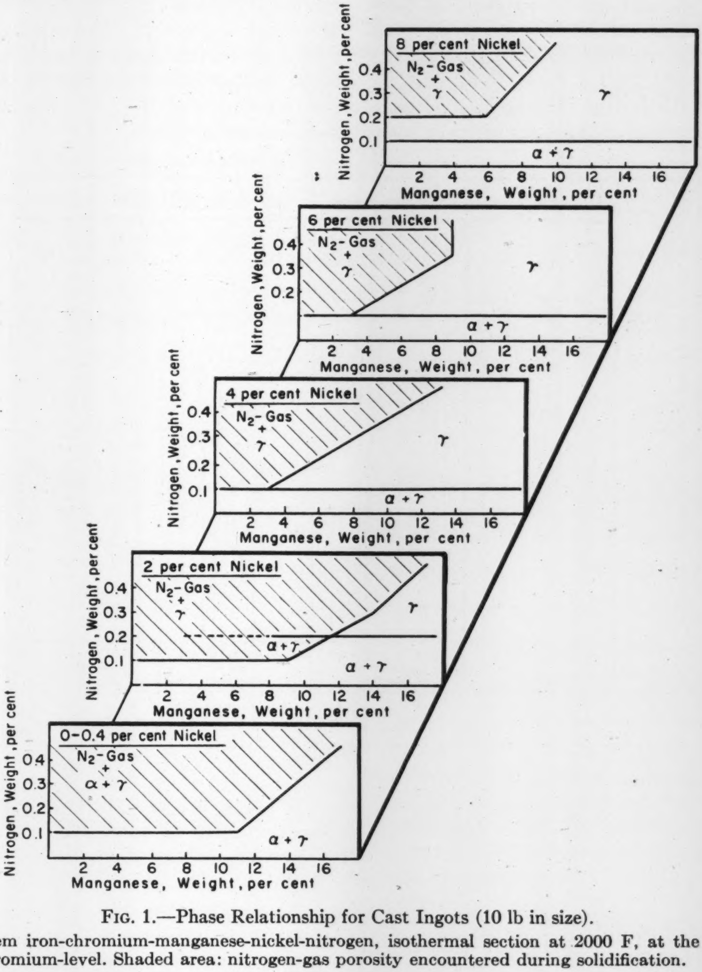


FIG. 1.—Phase Relationship for Cast Ingots (10 lb in size).

System iron-chromium-manganese-nickel-nitrogen, isothermal section at 2000 F, at the 18 per cent chromium-level. Shaded area: nitrogen-gas porosity encountered during solidification.

Phase Relations:

Austenite to austenite-plus-ferrite phase boundaries are shown in Fig. 1 for heat-treated cast compositions. They indicated that increasing the amount of nickel from 0.4 per cent causes the phase boundary to shift to lower nitrogen contents.

Forging and heat treatment at 1700 and 2000 F of austenitic-ferritic steel compositions effected a decrease in ferrite content. A 2300 F heat treatment increased the ferrite content in steel compositions containing low nitrogen contents. Wholly austenitic structures were shown to be attainable in the cast, forged, and heat-treated condition at the 2 to 8 per cent nickel levels, and the forged and 2000 F solution-treated condition of the 0 to 0.4 per cent nickel level. However, no fully austenitic structures could be attained at the 0 to 0.4 per cent nickel level in the as-cast and solution treated conditions.

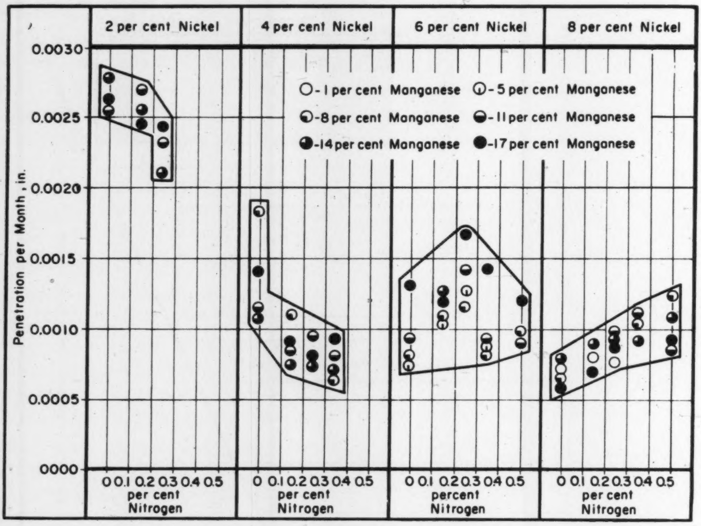


FIG. 2.—Huey Test: Average of 5 Periods of 48 hr Each in 65 per cent Boiling HNO_3.

Gas-Free Austenitic Compositional Ranges:

Figure 1 combines data obtained from the nitrogen solubility study and the austenite to ferrite relationships. The existence of fully austenitic gas-free compositional ranges are shown at the 2, 4, 6 and 8 per cent nickel levels. The unique properties of increasing amounts of nickel to extend the austenite-plus-ferrite to austenite phase boundaries to lower nitrogen contents and to increase the nitrogen solubility to higher nitrogen contents are noted.

Forgeability:

All compositions shown in Table I were found to be forgeable in the temperature range of 2100 to 1650 F.

It was noted that the high-nitrogen steels forged less readily and necessitated more reheating cycles. The high-nitrogen steels and low-nitrogen steels containing 4 per cent nickel and over showed increasing resistance to scale formation.

No difficulties were experienced in forging ingots containing nitrogen gas. Subsequent micro examination revealed that the voids present in the cast structures containing excessive nitrogen had been closed during the forging process.

Huey test results of some nickel-containing alloys are shown in Fig. 2.

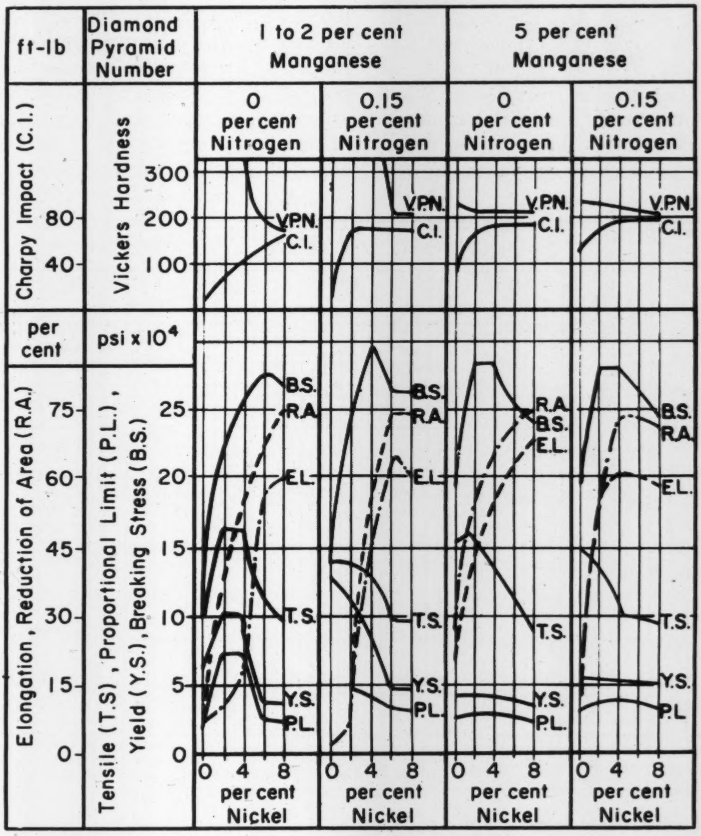


FIG. 3.—Mechanical Properties of Steels Containing 18 per cent Chromium, 1 to 2 and 5 per cent Manganese at 0, and 0.15 per cent Nitrogen Levels with Nickel Increasing from 0 to 8 per cent.

Corrosion Results:

Limited testing was conducted on nickel-free compositions. Huey test results of alloys containing 18 per cent chromium, 15 to 18 per cent manganese, and 0.2 to 0.5 per cent nitrogen were between 0.0030 to 0.0025 in. penetration per month.

In analyzing the influence of increasing amounts of manganese, nickel, and nitrogen, the following becomes apparent:

(*a*) Manganese appears to have little or no effect; (*b*) nickel increases the corrosion resistance and exhibits a marked influence at 4 per cent and (*c*) nitrogen shows a dual effect in that it increases the corrosion resistance at the 2 and 4 per cent nickel levels and lowers it at the 6 and 8 per cent nickel levels.

In an attempt to explain this dual behavior, it appears that as long as the ability of nitrogen to form austenite can be fully utilized, an improvement in corrosion resistance is realized. When alloyed into already austenitic structures it appears to lower the corrosion resistance. No evidence of intergranular corrosion was experienced in the Strauss test in samples having either ferritic-austenitic or austenitic structures.

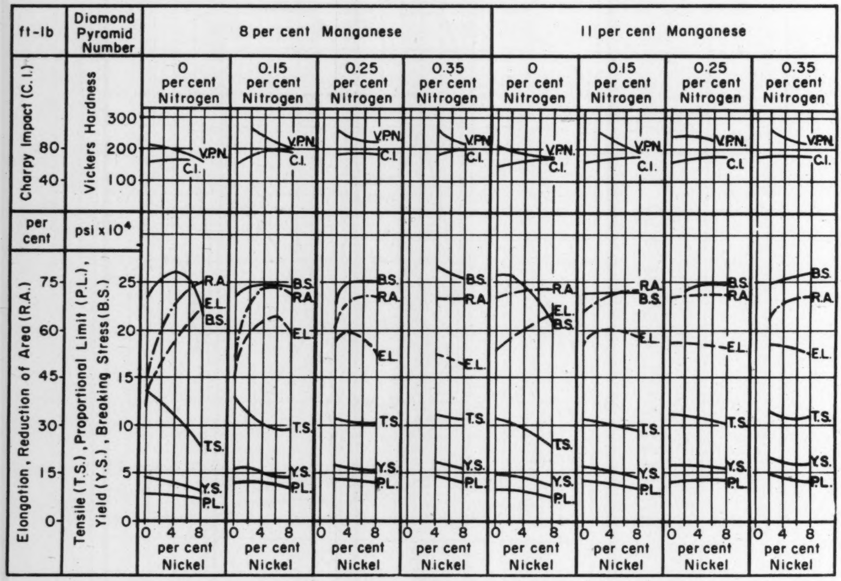


FIG. 4.—Mechanical Properties of Steels Containing 18 per cent Chromium, 8 and 11 per cent Manganese at 0, 0.15, 0.25, and 0.35 per cent Nitrogen Levels with Nickel Increasing from 0 to 8 per cent.

Mechanical Properties:

Mechanical property relationships are presented at various manganese levels and nitrogen sublevels with increasing nickel in Figs. 3, 4, and 5. The extreme changes in property values noted in Fig. 3 are due to changes from martensitic to ferritic-austenitic structures with increase in nickel. Figures 4 and 5 reflect ferritic-austenitic and wholly austenitic structures. These graphs indicate that property changes with increase in nickel content are influenced by the presence or absence of nitrogen. Increasing amounts of nickel in compositions containing no added nitrogen increases elongation and lowers tensile and yield strengths. However, in compositions containing 0.35 per cent nitrogen, elongation as well as tensile and yield strengths are lowered. Manganese exhibits little influence on mechanical property relationships. Increasing amounts of nitrogen increases tensile strength, yield strength, proportional limit, and hardness. Elongation and reduction of area are lowered whereas Charpy impact is little affected.

Embrittlement Studies:

The effects of embrittlement of two steel compositions containing 50 and 15 per cent delta-ferrite, respectively, on Charpy impact resistance are presented in the top two graphs of Fig. 6. The sharp drop in impact resistance with time at the 1000, 1200, 1400, and 1600 F levels was found to be associated with a transformation occurring within the delta-ferrite regions prior to the formation of sigma-phase or by sigma-phase formed from delta-ferrite. The 885 F embrittlement effect appears pronounced in the alloy containing 50 per cent delta-ferrite. With about 15 per cent delta-ferrite, no embrittlement was detected in 1000 hr at 800 F. This is in the range of the ferrite content required to meet minimum mechanical properties for the usual CF-8 castings. Embrittlement effects typical of chromium-manganese-nitrogen steels containing less than 5 per cent delta-ferrite are presented in the bottom graph of Figs. 6 and 7. These embrittlement effects are characterized by:

1. Grain boundary precipitation (Cr_4C) at the 1000 F level.

2. Grain boundary and lamellar precipitation (Cr_4C or $Cr_4C + Cr_2N$) at the 1200 and 1400 F levels.

3. Grain boundary precipitation (Cr_2N or $Cr_2N + Cr_4C$) at the 1600 F level.

4. No ferrite is formed as a consequence of Cr_2N or Cr_4C precipitation. The reactions have the nature of austenite →

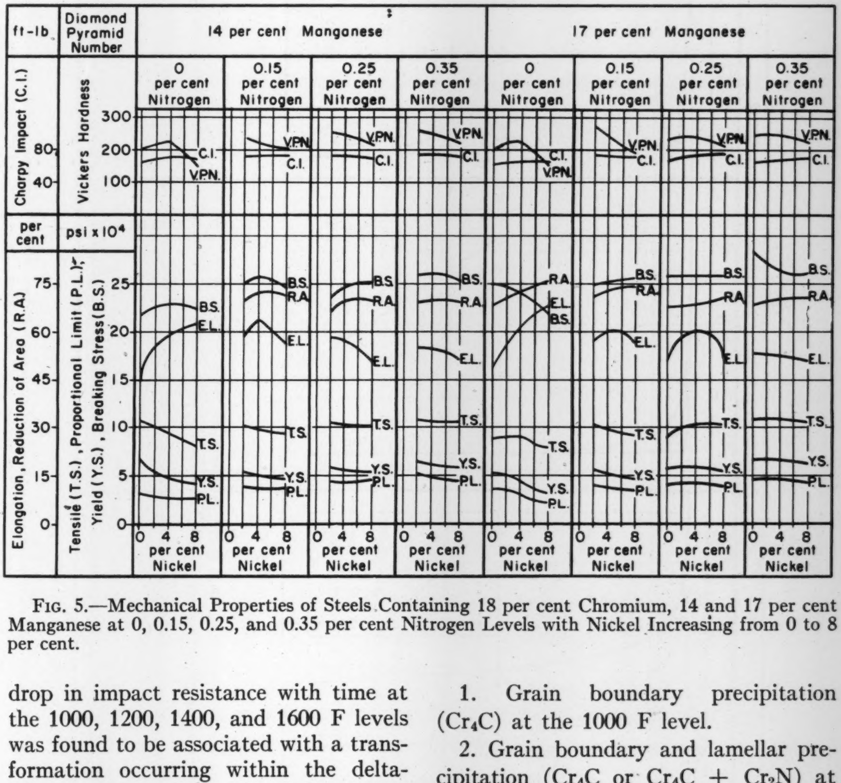


FIG. 5.—Mechanical Properties of Steels Containing 18 per cent Chromium, 14 and 17 per cent Manganese at 0, 0.15, 0.25, and 0.35 per cent Nitrogen Levels with Nickel Increasing from 0 to 8 per cent.

Cr_2N + austenite or austenite → Cr_4C + austenite (1).

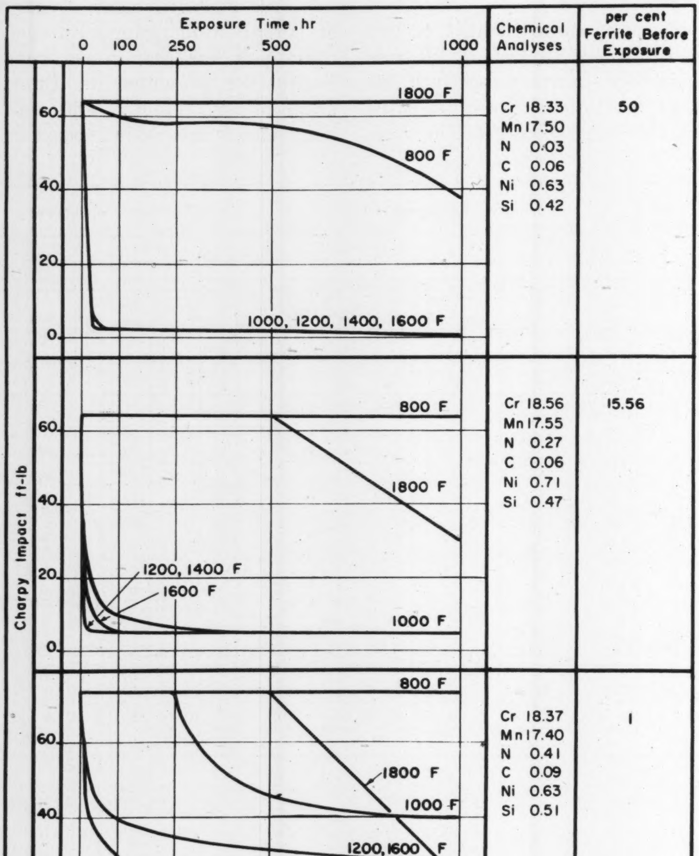


FIG. 6.—Showing Embrittlement of Austenitic-Ferritic Alloys.

The embrittlement occurring at 1800 F in steel compositions containing nitrogen appears to be of a complex nature. Heavy grain boundary as well as lamellar and Widmanstätten precipitation occur on the outer portions of the Charpy bars, surrounding a core of original austenite. X-ray diffraction studies indicate the presence of Cr_2N plus some unidentified lines. Hardness surveys

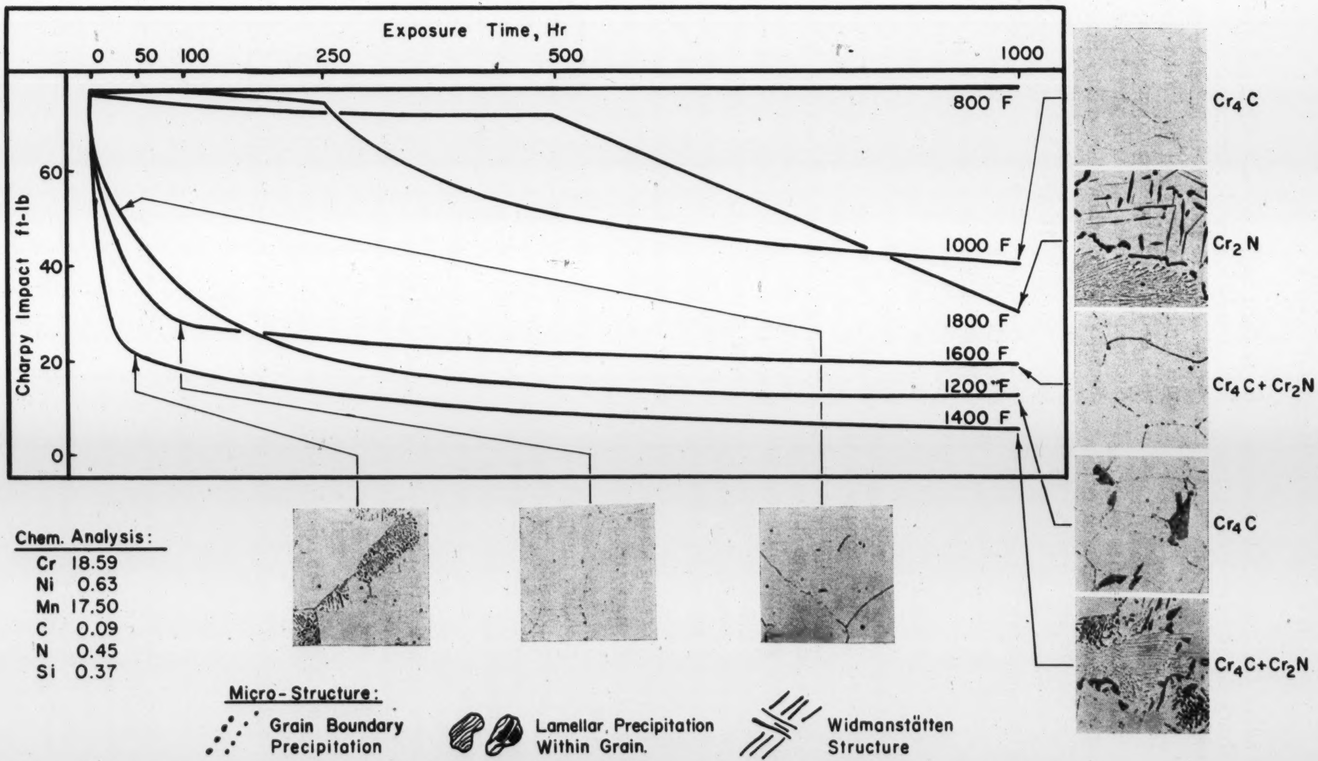


FIG. 7.—Showing Embrittlement of a Fully Austenitic Alloy.

used to follow Cr_4C and Cr_2N precipitation embrittlement have proven unreliable and are not presented.

Casting Studies:

Some sand casting studies were conducted within the austenitic castability ranges. The fluidity and pouring characteristics were found to be satisfactory. Sound 2-in. gate valve bodies were produced. Further casting evaluation is in progress.

SUMMARY

The following property relationships were established in steel compositions containing 18 per cent chromium, 0 to 18 per cent manganese, 0 to 8 per cent nickel, 0 to 0.5 per cent nitrogen and approximately 0.08 per cent carbon.

1. The elements manganese and nickel increase the nitrogen solid solubility limit.
2. The austenite-forming ability of nitrogen, nickel, and manganese decrease in order stated.
3. Gas-free austenitic compositional ranges are made possible by proper balance of the elements manganese, nickel, and nitrogen.
4. Forging and solution treatment at 2000 F reduce ferrite content.
5. Manganese has little effect on the corrosion resistance. Nickel increases corrosion resistance and shows a marked influence at 4 per cent and higher. Nitrogen exhibits a dual behavior in that it increases the corrosion resistance as long as its ability to form austenite can be utilized and lowers it when alloyed into already austenitic structures.
6. Manganese shows little influence on mechanical properties of austenitic-ferritic and austenitic steel compositions. Nitrogen increases strength and lowers ductility. Nickel when alloyed into nitrogen-free steels increases ductility and lowers strength. However, when alloyed into nitrogen-containing steels both strength and ductility are lowered.
7. Low-nickel compositions containing more than 5 per cent ferrite show pronounced embrittlement in the temperature ranges of 1000 to 1600 F. This is due to changes occurring within ferrite areas prior to sigma-phase formation and by sigma-phase formed from ferrite. With about 15 per cent ferrite no embrittlement was detected in 1000 hr at 800 F.
8. Highly austenitic steel compositions containing low nickel embrittle in the temperature range of 1000 to 1600 F due to grain boundary and lamellar precipitates, which are Cr_4C or Cr_2N or both.
9. Nitrogen-containing steels after long time annealing at 1800 F show a localized precipitation embrittlement.
10. Sand casting studies conducted within the austenitic castability ranges indicate promise.

Acknowledgment:

The authors express their thanks to the members of the Crane Research Laboratories who helped in carrying out this project. Appreciation is also extended to The Standard Oil Co. of Indiana for X-ray diffraction studies.

REFERENCES

(1) W. Koester, "Die Eisenecke des Systems Eisen-Mangan-Chrom," *Archiv für das Eisenhüttenwesen,* Vol. 7, pp. 687–688 (1933–1934).

(2) F. Bruehl, "Gefüge und Eigenschaften von Chrom-Mangan-Stählen, etc.," *Archiv für das Eisenhüttenwesen,* Vol. 10, pp. 243–255 (1936–1937).

(3) P. Schafmeister und R. Ergang, "Die spröde O-Phase im Fe-Cr-Mn Zustandschaubild," *Archiv für das Eisenhüttenwesen,* Vol. 12, pp. 507–510 (1938–1939).

(4) H. Krainer, "Gefüge, Härte, Kerbschlagzähigkeit und Magnetische Sättigung Hitzebeständiger Chrom-Mangan-Stahle," *Archiv für das Eisenhüttenwesen*, Vol. 28, pp. 81–89 (1957).

(5) E. Houdremont, Handbuch der Sonderstahlkunde, Springer Verlag, pp. 706–727, 1327–1330 (1956).

(6) R. Franks, W. D. Binder, and T. Thompson, "Austenitic Chromium-Manganese-Nickel Steels Containing Nitrogen," *Preprint No. 29*, Am. Soc. Metals, Vol. 47 (1954).

(7) W. R. Renshaw and R. A. Lula, "The Corrosion Properties of Chromium-Nickel-Manganese Austenitic Stainless Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 866 (1956).

(8) D. J. Carney, "Nickel Free and Low Nickel Austenitic Stainless Steels," presented at Cleveland Regional Technical Meeting of Am. Iron and Steel Inst., Oct. 11, 1955.

(9) V. F. Zackay, J. F. Carlson, and P. L. Jackson, "High Nitrogen Austenitic Cr-Mn-Steels," *Preprint No. 5*, Am. Soc. Metals, Vol. 48 (1955).

(10) Chi-Mei Hsiao and E. J. Dulis, "Precipitation Reactions in Austenitic Cr-Mn-N Stainless Steels," *Preprint No. 26*, Am. Soc. Metals, Vol. 49 (1956).

DISCUSSION

MR. F. C. HULL.[1]—Are these materials intended for welding and have any welding tests been conducted on them? What maximum temperature of operation is recommended for them for long-time service?

MR. J. J. KANTER (*author*).—Of course, these questions would have a different answer for each of the compositions. If you are discussing compositions in which austenite is rather heavily stabilized with nickel, one could conclude that the welding properties are about the same as for chromium-nickel austenitic steels. With less stability and higher ferrite content the welding problem increases. Limited welding tests made on the 4 per cent nickel group showed acceptable weldability. Similar wrought compositions were shown to have adequate weldability by Franks, Binder and Thompson.[2]

With regard to high temperature properties, tests were conducted on the 0.4 per cent nickel series. The fully austenitic high-nitrogen steels compare favorably with commercial austenitic steels from the standpoint of tensile and creep strength at temperatures up to 900 F. Some loss in ductility and impact resistance occurs in these alloys containing less than about 0.3 per cent nitrogen and larger amounts of ferrite in the microstructure.

A paper published by Allegheny Ludlum Steel Corp.[3] indicates that the high-temperature strengths of similar 4 to 6 per cent nickel compositions are slightly better than those of type 302 steels.

---

[1] Advisory Metallurgist, Westinghouse Research Laboratories, Metallurgy Department, Westinghouse Electric Corp., Pittsburgh, Pa.

[2] *Transactions*, Am. Soc. Metals, Vol. 47, p. 231 (1955).

[3] *Publication No. 3*, Technical Studies (1956).

EFFECT OF COLD ROLLING AND STRESS RELIEF ON THE SHARP EDGE NOTCH TENSILE CHARACTERISTICS OF AUSTENITIC STAINLESS STEEL SHEET ALLOYS*

By G. B. Espey, A. J. Repko, and W. F. Brown, Jr.[1]

Synopsis

Smooth and sharp edge notch tensile characteristics are established at room temperature and −320 F for cold-rolled types AISI 301 and AISI 304 L steel sheet. Reductions by room temperature rolling varied from 20 to 70 per cent, to a final constant gage of 0.063. Stress relief treatments were investigated for type AISI 301 steel rolled to 60 per cent reduction and type AISI 304 L steel rolled to 70 per cent reduction. Temperatures were from 400 to 1100 F; times were 24 and 72 hr.

At room temperature, relatively high values of sharp notch-yield strength ratios characterizes both steels tested in the longitudinal direction. In contrast, high transverse notch sensitivity is observed for both alloys cold-worked more than about 40 per cent. At −320 F the longitudinal direction retains relatively high notch toughness while the transverse notch sensitivity is increased, particularly for type AISI 301.

Stress relief for 24 hr in the temperature range between 400 and 700 F raises the room temperature smooth tensile and yield strength of both alloys. This beneficial effect is less pronounced for tests at −320 F. Generally, the effects of stress relief on the notch sensitivity are quite complex and not entirely accounted for by known metallurgical influences. No appreciable reduction in notch sensitivity is produced by any of the stress relief treatments Stress relief at temperatures above 900 F produces considerably increased −320 F notch sensitivity in both alloys. An interesting observation is the development of pronounced embrittlement in type 304 L steel when stress relieved in the temperature range between 600 and 800 F.

Casing materials for advanced solid and liquid propellant rockets require yield strengths in excess of 200,000 psi plus high notch toughness, for different ranges of use temperature. Recent investigations (**1**,**2**,**3**)[2] have shown that when ferritic steels are heat treated to approximately this high strength level, many compositions and heat treatments are subject to brittle fracture at room temperature. Above this strength level, most martensitic steels are highly sensitive to stress concentrations, such as may be created by flaws, impurities, scratches and cracks. Thus, catastrophic fractures can occur at very low loads in both laboratory specimens and in hardware.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Lewis Research Center, National Aeronautics and Space Administration, Cleveland, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Austenitic stainless steels have been used for many years at very low temperatures, in applications where high toughness is of particular advantage. Relatively high conventional impact values (4,5) at low temperatures suggest that these steels may possess high notch toughness in the presence of severe stress concentrations, in contrast to the ferritic alloys. However, high yield strengths have not been of primary concern in past development and application of austenitic stainless steels. These alloys have been used primarily in the annealed condition or with moderate amounts of cold work. To meet the high yield strength requirements for advanced missiles, much more "cold" strengthening is required.

Only limited data is available for these alloys at the high deformations necessary to produce high strength levels. Krivobok and Talbot (6) investigated the metastable austenitic alloy AISI type 301. For a rolling reduction of 60 per cent, they report yield strengths over 200,000 psi when deformation was at room temperature and over 300,000 psi when deformation was at −105 F. As pointed out by Binder (7) and by Ziegler and Brace (8), lower temperatures promote the $\nu \longrightarrow \alpha$ transformation and increase the strengthening per unit of plastic deformation. Because this strengthening is strongly dependent on the martensite formed, the nickel and carbon content as well as the deformation temperature and speed, are major variables influencing the final properties. The elongations of these severely cold-worked steels are only a few per cent. This in itself need not necessarily prohibit their use in large cylindrical pressure vessels, if special methods of fabrication are employed.

On the other hand, there appear to be several additional factors which may be pertinent to the application of cold-worked austenitic stainless steels, namely:

(*a*) The increase in directionality of mechanical properties with increasing cold reduction. Franks and Binder (9,10) have observed directionality in the tensile ultimate and yield strength of several AISI 300 steel types cold worked up to 50 per cent. This directionality appears to increase with the amount of cold reduction (4).

(*b*) The possibility of notch of sensitivity in metastable austenite at high cold reductions, particularly at low temperatures.

(*c*) The degree to which the smooth tensile properties are influenced by stress relief heat treatment and the corrosion resistance of stress relieved steel. It has been known for many years (11,12) that the tensile yield strength of moderately cold-worked stainless steels is raised by stress relief heat treatment. On the other hand, as pointed out by Franks and Binder (9) care should be taken that the stress relief treatment does not "sensitize" the steel, through carbide precipitation. It may be inferred from the results of Mahla and Nielsen (13) that the tendency to sensitization will decrease with increasing cold work. This may be explained by a more uniform carbide distribution in the cold-worked steels and the "keying" effect of the deformed grains.

(*d*) The possible deterioration in notch toughness caused by heating the steel in a range of temperatures producing carbide precipitation. Schmidt (14) and Krivobok (15) report the low temperature impact values of moderately cold-worked steels are drastically reduced by heating 2 hr at 1200 F. This effect appears to increase with the amount of cold reduction. In a recent investigation, Kramer and Baldwin (16) report the tensile ductility of sensitized AISI types 304 and 302 steels decreases with a decrease in test temperature and strain rate.

The present investigation is an attempt to develop further information relating to the above problems. Two sheet alloys were selected, the metastable AISI type 301 and the presumably stable AISI type 304 L. The sharp edge notch tensile test (3) was used to assess the fracture toughness of these alloys as a function of cold reduction (up to 70 per cent) at both room temperature and −320 F. In addition, the effect of stress relief heat treatments on the sharp notch properties were investigated for one severely cold-worked condition of each alloy. The corrosion rates in boiling 65 per cent nitric acid were also established as a function of the stress relief temperature and time.

TABLE I.—CHEMICAL COMPOSITION OF INVESTIGATED STEELS.[a]

AISI Type	Heat Number	Composition, per cent						
		Carbon	Manganese	Phosphorus	Sulfur	Silicon	Nickel	Chromium
301	9X9343	0.10	1.24	0.033	0.020	0.53	7.28	17.16
304 L	7X1964	0.017	1.38	0.016	0.019	0.65	9.44	18.18

[a] Steels furnished by Am. Steel and Wire Division, U. S. Steel Corp.

MATERIAL AND PROCEDURE

The composition of the two alloys investigated is given in Table I. Both steels were obtained in 0.063-in. thickness and in several degrees of cold-reduction up to 70 per cent.

The sharp-notch and smooth tensile specimens are shown in Fig. 1. Details regarding selection of these particular specimen types and techniques of machining have been discussed previously (3). Specimens were completely machined and degreased before stress relieving. Stress relief was carried out in an air circulating furnace at a series of temperatures from 400 F to 1100 F.

Room temperature tests employed fixtures and techniques already described (3). The −320 F data were obtained using essentially the same fixtures, the specimen and grips being surrounded by an insulated stainless steel can containing liquid nitrogen. This can was provided with a Teflon packing gland which permitted it to be moved upward and downward on the lower loading rod in order to allow installation and removal of specimens. A specially constructed extensometer was used to measure the longitudinal strain of smooth specimens over a 2-in. gage length at −320 F. The sensitivity of strain measurement was 0.020 per cent. The use of this instrument required that small drill (No. 60) marks be placed in the surface on one side of the specimen. In some cases fracture occurred through these marks. In order to determine reliable ultimate tensile strengths, dupli-

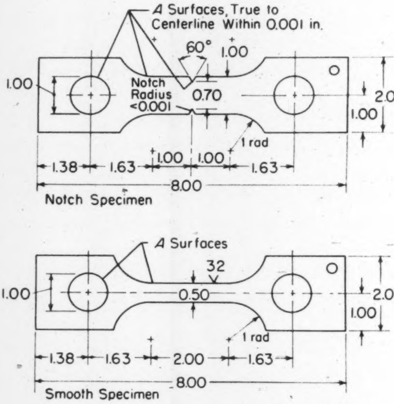


FIG. 1.—Smooth and Sharp-Edge-Notch Sheet Tensile Specimens.

cate tests without drill marks were run for these conditions. Elongations in 2 in. were measured between lightly scribed marks.

The corrosion studies (see Appendix) were carried out by the Applied Research Laboratory of the U.S. Steel Corp.

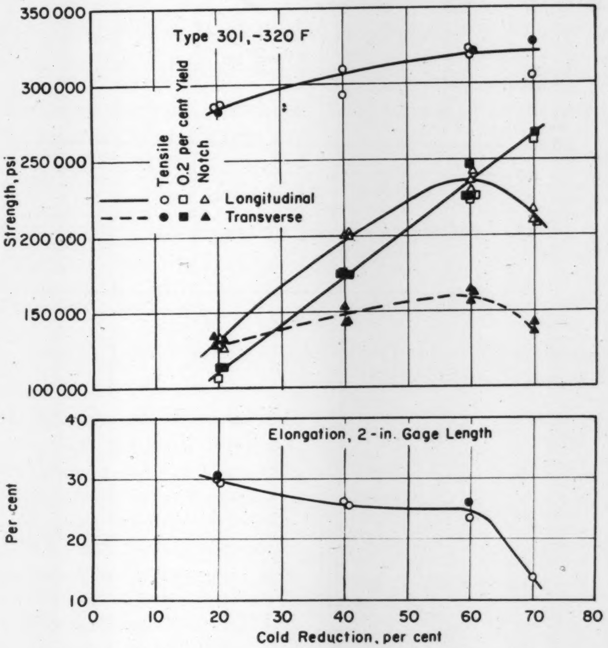


FIG. 2.—Sharp-Notch and Smooth Tensile Characteristics at −320 F for 0.063-in. AISI Type 301 Sheet as a Function of Cold Reduction.

DISCUSSION OF THE EFFECTS OF COLD REDUCTION

Room temperature sharp-notch and smooth tensile data for both AISI type 301 and 304 L steels have been previously reported (3). These data covered cold reductions from 20 to 70 per cent for AISI type 301 and from 40 to 70 per cent for AISI type 304 L. These tests were repeated at −320 F (Figs. 2 and 3). The following section will consider the effect of this low temperature on both the smooth and notch properties of these cold-rolled conditions without stress relief heat treatments.

Smooth Tensile Properties:

The ratio between the smooth tensile and yield strength at −320 F to these strength values at room temperature is shown in Fig. 4 as a function of cold reduction. Neglecting small differences between longitudinal and transverse properties both alloys may be represented by the same curves to close approximation. As might be expected both the tensile and yield strengths are raised by testing at low temperature. The elevation is much

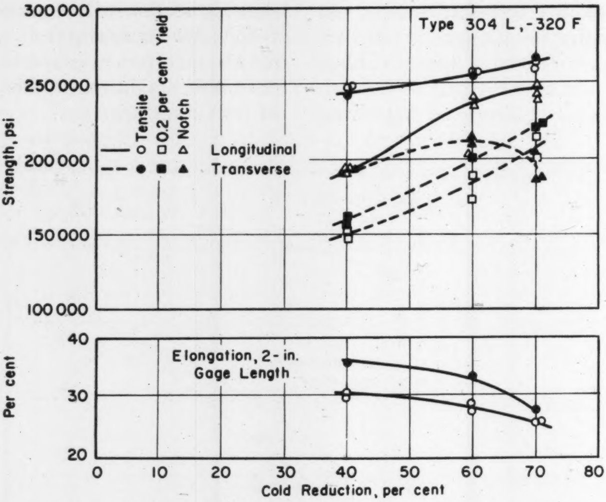


FIG. 3.—Sharp-Notch and Smooth Tensile Characteristics at −320 F for 0.063-in. AISI Type 304 L Sheet as a Function of Cold Reduction.

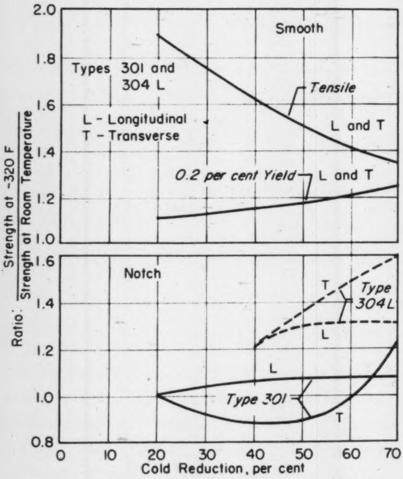


FIG. 4.—Ratio Between the Strength at −320 F to the Strength at Room Temperature as a Function of Cold-Reduction for AISI Type 301 and 304 L 0.063-in. Sheet.

larger for the tensile than for the yield strength and depends strongly on the amount of prior cold reduction. This behavior results in very low yield strength to tensile strength ratios at −320 F for the lower values of cold reduction (for example, 0.4 for AISI type 301 at 20 per cent cold-reduction, Fig. 2). The elongations at −320 F (Figs. 2 and 3) are high for both steels. The values decrease with increasing cold reduction and for type AISI 301 an abrupt drop occurs between 60 and 70 per cent.

The above described phenomena have been previously reported by other investigators (**6,15**). These results serve to emphasize that the behavior of austenitic stainless steels at cryogenic temperatures is much different from that observed for ferritic alloys tested at room temperature or low temperature.

Notch Properties:

The influence of low temperature on the sharp-notch strength,[3] (Fig. 4) is quite different from that observed for the smooth strength. Furthermore, distinct differences in behavior are observed between the longitudinal and transverse directions. For AISI type 301 the −320 F longitudinal sharp-notch strengths are only slightly higher than the room temperature values. In the transverse direction, the notch strengths at −320 F appear to be somewhat lower than those at room temperature in the intermediate range of cold reductions but higher than the room temperature values at 70 per cent cold reduction. For AISI type 304 L the longitudinal and transverse notch strengths are higher at −320 F than at room temperature. This difference increases with the amount of cold reduction, particularly for transverse tests.

---

[3] The notch strength and tensile strength are determined by dividing the maximum load by the initial minimum area.

Effects of Cold-Reduction on Notch Sensitivity:

The notch sensitivity of a given alloy condition is frequently expressed either as the ratio between the notch strength and the tensile strength or between the notch strength and the yield strength. The former ratio has been generally referred to as the "sharp notch strength ratio" and this terminology will be followed here. The latter ratio is used less frequently but may have some practical advantage where components are designed to a yield strength limit. It will be designated as the "sharp notch yield strength ratio." In heat-treated alloy steels a low sharp-notch strength ratio always characterizes brittle failure of notched sheet specimens at stresses well below the yield strength (3). Furthermore, completely notch ductile behavior is characterized by sharp-notch strength ratios of unity or slightly above. For such steels the sharp-notch strength ratio is a satisfactory indicator of relative performance in the presence of high stress concentrations.

For the present alloys, entirely different fracture behaviors are noted, particularly where the yield strength to tensile strength ratios are very low. Under these conditions the sharp-notch strength ratio may be substantially below unity while the notch strength is still above the yield strength (for example, AISI type 301 longitudinal tests at −320 F, Fig. 2). Where this behavior is observed, the notch specimen exhibits a jagged fracture. Cracking at the notch root occurs at low loads and is probably aided by localized martensite formation associated with the high plastic strain gradient at the notch root. The crack runs in an irregular path with high plastic strains occurring in the uncracked areas. For steels exhibiting this behavior low values of the sharp-notch strength ratio indicate the full tensile strength potential cannot be realized in the presence of high stress concentrations but do not necessarily indicate brittle fracture.

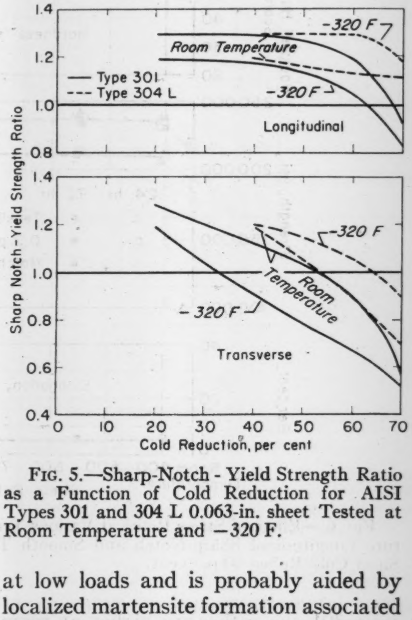


FIG. 5.—Sharp-Notch - Yield Strength Ratio as a Function of Cold Reduction for AISI Types 301 and 304 L 0.063-in. sheet Tested at Room Temperature and −320 F.

The sharp notch yield strength ratio is shown in Fig. 5 as a function of cold-reduction for both steels tested at room temperature and −320 F. Several important observations can be made from this representation: (*a*) The sharp-notch to yield strength ratios of both steels decrease with increasing cold-reduction. This effect is most pronounced for tests in the transverse direction. (*b*) For AISI type 301 the ratios are higher at room than at −320 F. In contrast, for AISI type 304 L a reverse effect of testing temperature is observed. (*c*) In the longitudinal direction, ratios less than unity are obtained only at cold-reductions over about 60 per cent. (*d*) In the transverse direction much lower values are noted. At room temperature ratios less than unity are observed for both alloys cold-reduced more than 50 per cent.

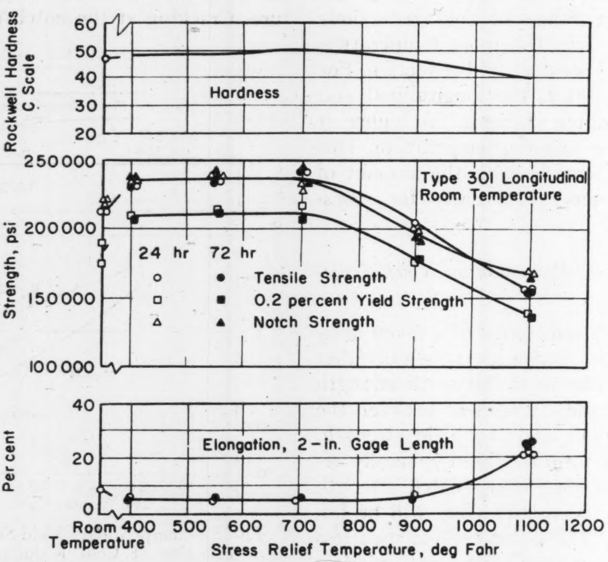


FIG. 6.—Effect of Stress Relief at Various Temperatures for 24 and 72 hr on the Room Temperature Longitudinal Sharp-Notch and Smooth Tensile Characteristics of 0.063-in. AISI Type 301 Sheet Cold-Rolled 60 per cent.

DISCUSSION OF THE EFFECT OF STRESS RELIEF HEAT TREATMENT

The phase of the investigation next discussed was confined to the highest cold-reduction producing a longitudinal sharp notch to yield strength ratio of unity or above in each alloy. This corresponds to 60 per cent cold-reduction for AISI type 301 and 70 per cent cold-reduction for AISI type 304 L (see Fig. 5).

From the results of Franks and Binder (9) stress relief times of 24 and 72 hr were selected. Long times at low temperature apparently permit stress relief without introducing appreciable carbide precipitations. In this investigation the temperature range extended from 400 F to 1100 F in order to explore the possible effect of precipitation on both the sharp-notch strength and corrosion behavior.

In addition, corrosion samples were stress relieved 4 hr at 1200 F, a condition known to produce "sensitization" in annealed 18-8 steels.

The primary sharp-notch and smooth tensile data is shown in Figs. 6 to 13 as a function of stress relief temperature. It will be noted that there is essentially no difference between the 24 and 72 hr treatments. Rather complex behaviors are observed particularly in the case of the sharp-notch strength. All the observed phenomena are difficult to completely explain without detailed structural studies. However, the observed effects may be related in part to the interaction of several factors: (*a*) reduction of residual stresses, (*b*) softening of the heavily cold-worked structure, (*c*) precipitation of carbides in grain boundaries and slip planes,[4] and (*d*) formation of martensite during testing. On the basis of the present results it is not possible to give a detailed description of how these various phenomena influence the strength behavior. However, in some cases it does appear possible to decide which mechanisms are dominant in a given range of the experimental variables.

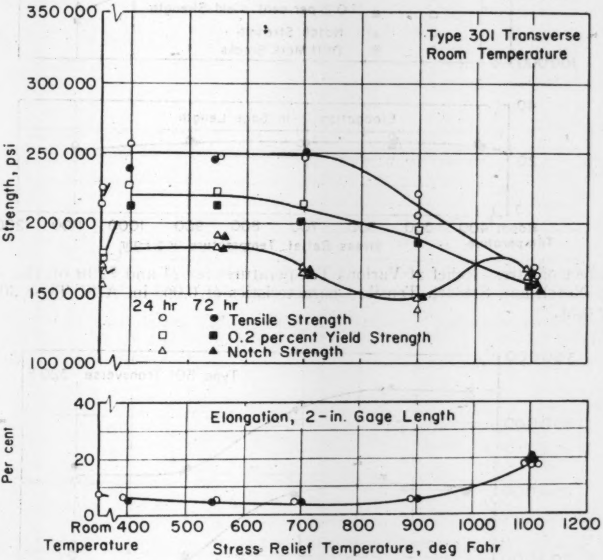


FIG. 7.—Effect of Stress Relief at Various Temperatures for 24 and 72 hr on the Room Temperature Transverse Sharp-Notch and Smooth Tensile Characteristics of 0.063-in. AISI Type 301 Sheet Cold-Rolled 60 per cent.

Hardness:

The room temperature hardness is shown as a function of the stress relief temperature in Figs. 6 and 10 for AISI type 301 and AISI type 304 L, respec-

---

[4] The precipitation of carbides may result in some ferrite formation; however, this is considered to have a negligible effect on the mechanical properties of these heavily cold-worked alloys.

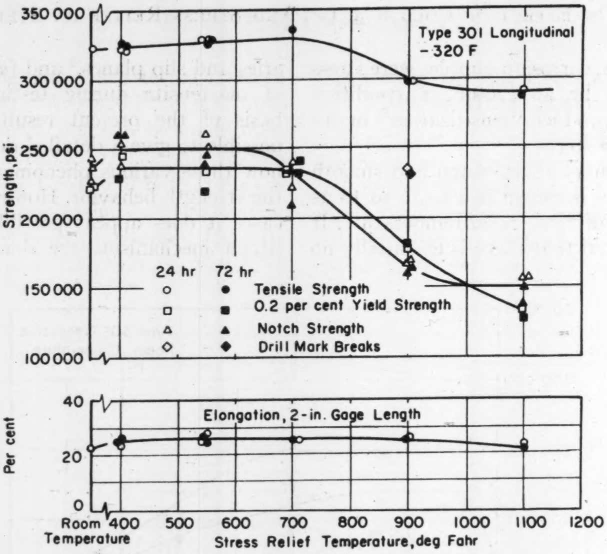


FIG. 8.—Effect of Stress Relief at Various Temperatures for 24 and 72 hr on the −320 F Longitudinal Sharp-Notch and Smooth Tensile Characteristics of 0.063-in. AISI Type 301 Sheet Cold-Rolled 60 per cent.

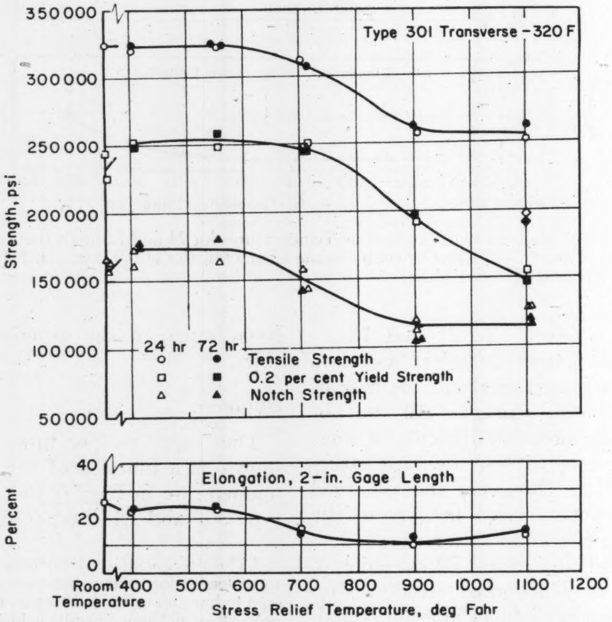


FIG. 9.—Effect of Stress Relief at Various Temperatures for 24 and 72 hr on the −320 F Transverse Sharp-Notch and Smooth Tensile Characteristics of 0.063-in. AISI Type 301 Sheet Cold-Rolled 60 per cent.

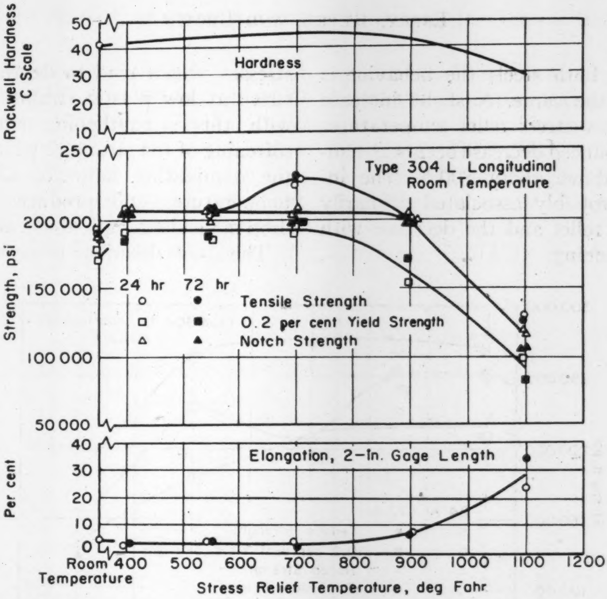


FIG. 10.—Effect of Stress Relief at Various Temperatures for 24 and 72 hr on the Room Temperature Longitudinal Sharp-Notch and Smooth Tensile Characteristics of 0.063-in. AISI Type 304 L Sheet Cold-Rolled 70 per cent.

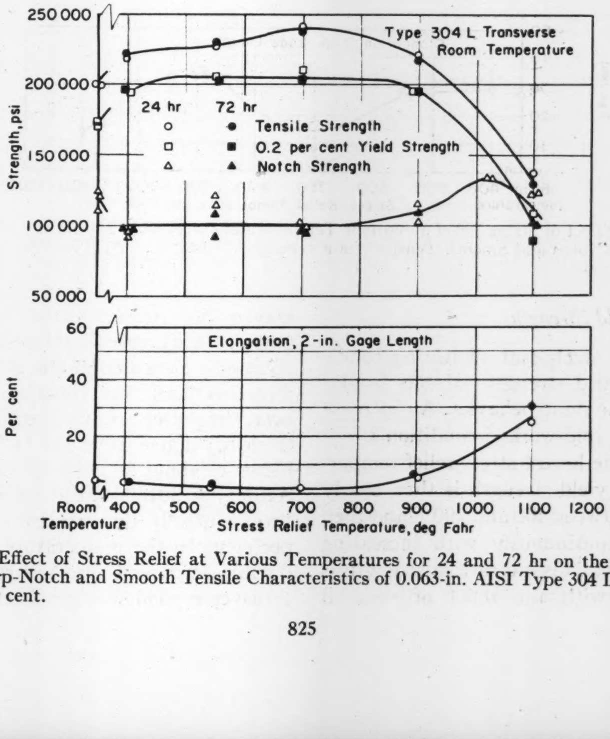


FIG. 11.—Effect of Stress Relief at Various Temperatures for 24 and 72 hr on the Room Temperature Sharp-Notch and Smooth Tensile Characteristics of 0.063-in. AISI Type 304 L Sheet Cold-Rolled 70 per cent.

tively. For both steels the behavior is essentially the same. A slight increase occurs at low stress relief temperatures and a pronounced decrease occurs at temperatures above about 800 F. The increase is probably associated primarily with stress relief and the decrease with general softening.

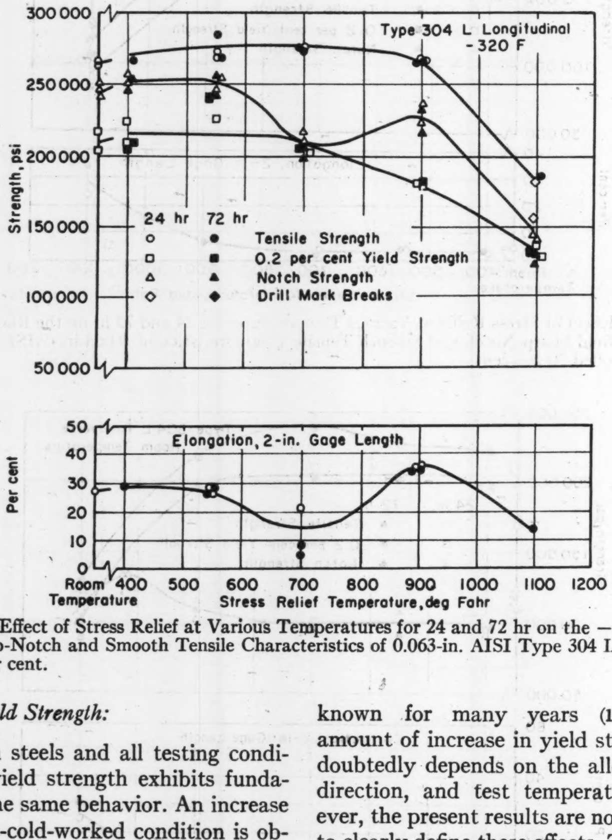


FIG. 12.—Effect of Stress Relief at Various Temperatures for 24 and 72 hr on the −320 F Longitudinal Sharp-Notch and Smooth Tensile Characteristics of 0.063-in. AISI Type 304 L Sheet Cold-Rolled 70 per cent.

Tensile Yield Strength:

For both steels and all testing conditions the yield strength exhibits fundamentally the same behavior. An increase over the as-cold-worked condition is observed at the lowest stress relief temperature. The yield strength is then nearly constant between 400 and 700 F and then decreases continuously with increasing temperature. This increase in strength is associated with the relief of residual stresses, which tend to depress the flow curve at low plastic strains. Combined with this strengthening effect is the softening of the structure which becomes the dominating influence at the high temperatures and produces the rapid drop in yield strength above about 700 F.

The above described behavior has been known for many years (11,12). The amount of increase in yield strength undoubtedly depends on the alloy, testing direction, and test temperature. However, the present results are not sufficient to clearly define these effects. The largest increase (about 20 per cent) occurred for AISI type 301 tested at room temperature, generally the increase was 5 to 10 per cent. In the temperature range producing improved yield strengths the transverse yield was consistently higher than the longitudinal yield by about 5 per cent for AISI type 301 and about 10 per cent for AISI type 304 L. These differences tend to fade out at the higher stress relief temperatures. This is in contrast to the behavior of the alloys without stress relief which with the exception of AISI type 304 L (room temperature) showed nearly identical transverse and longitudinal yield values.

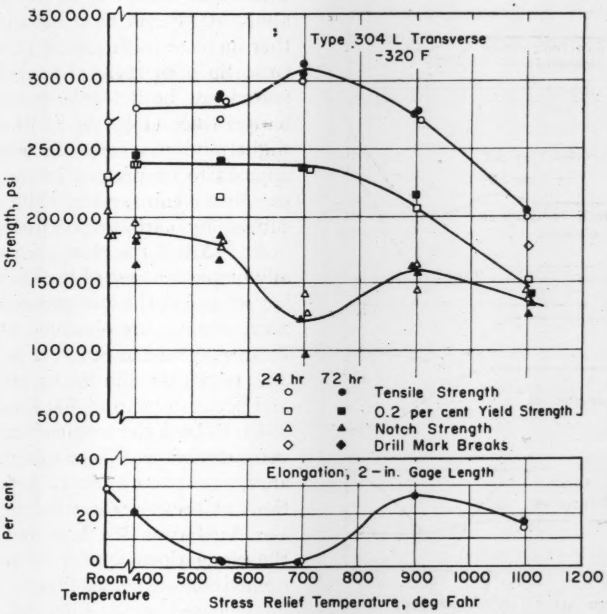


FIG. 13.—Effect of Stress Relief at Various Temperatures for 24 and 72 hr on the −320 F Transverse Sharp-Notch and Smooth Tensile Characteristics of 0.063-in. AISI Type 304 L Sheet Cold-Rolled 70 per cent.

Tensile Strength:

An initial elevation of tensile strength at 400 F is observed in both steels tested at room temperature (Figs. 6 and 7, and Figs. 10 and 11). This trend parallels that of the yield strength and is associated with the very low necking strain characterizing these heavily cold-worked conditions. This low necking strain permits the retention of some residual stresses up to maximum load. In contrast, at −320 F (Figs. 8, 9, 12, and 13) no corresponding elevation of the tensile strength is observed. This may be explained by the fact that the necking strain at −320 F is considerably higher and plastic flow is available to wipe out a large portion of the residual stresses present in the as-cold-worked steels. If softening were the predominate behavior at the higher stress relief temperatures, then the tensile strength should follow essentially the yield strength trend. Such basic behavior is observed for AISI type 301 (Figs. 6 and 7) tested at room temperature. However, the same steel tested at −320 F exhibits nearly constant tensile strength between 900 and 1100 F stress relief temperatures. This may be explained by the tendency of these softer conditions to produce more martensite during testing at −320 F.

For AISI type 304 L the tensile strength exhibits a slight maximum at 700 F which appears to be more pronounced for tests at room temperature than at −320 F. Heating at 700 F may produce strengthening by carbide precipitation and by conditioning the austenite for subsequent transformation, during testing, to a very low carbon martensite. At high stress-relief temperatures the tensile strength of AISI type 304 L decreases continuously, the dominant mechanism being one of softening.

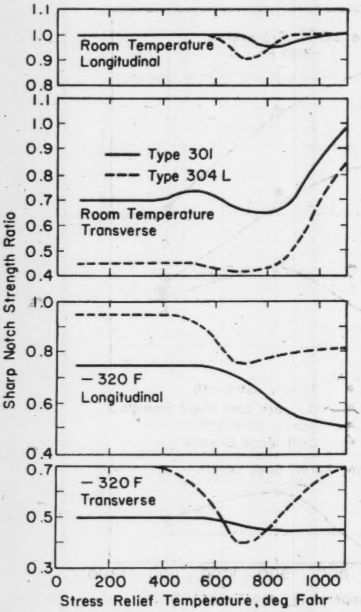


FIG. 14.—Sharp-Notch - Strength Ratio at Room Temperature and −320 F for AISI Types 301 and 304 L Cold-Rolled 0.063-in. Sheet Stress Relieved at Various Temperatures.

Elongations:

The room temperature elongations of the as-cold-rolled alloys are in the order of 10 per cent for AISI type 301 and 5 per cent for AISI type 304 L. If softening were the only effect of the stress relief heat treatment the elongation should remain essentially equal to that of the cold-rolled alloy to a temperature of about 700 F and then increase with further increase in the stress relief temperature. Such behavior is closely approximated by both steels tested at room temperature (Figs. 6, 7, 10, and 11). A slight minimum between 600 and 800 F appears to be present in the room temperature transverse values possibly caused by carbide precipitation.

At −320 F the elongations are generally higher because of the increased necking strains at the low temperature. However, minima are observed at about 900 F stress relief temperature for AISI type 301 tested in the transverse direction and between 500 and 800 F for AISI type 304 L in both the longitudinal and transverse directions. These minima are probably associated with carbide precipitations at these stress relief temperatures. For AISI type 304 L tested at −320 F the elongations appear to pass through a maximum at about 900 F. This behavior is based on only a few data points and may not be real.

Notch Properties:

The sharp-notch strength ratio has been plotted against the stress relief temperature in Fig. 14. If stress relief and softening were the dominant mechanism in determining the notch strength, the sharp-notch strength ratio would be essentially constant over the entire range of stress relief temperatures for an alloy notch ductile in the as-cold-worked condition. However, shallow minima are observed at stress relief temperatures between 600 and 800 F for both steels tested in the longitudinal direction at room temperature. The room temperature transverse sharp-notch strength ratio for the as-cold-worked alloys is low. In the absence of other factors this ratio would be expected to continuously increase with increased softening. However, pronounced minima are observed for both steels in the temperature range from 600 to 800 F. These correspond to the smaller minima observed for longitudinal room temperature tests.

It is considered that carbide precipitation is associated with the above described behaviors. In this connection, it should be noted that a maximum in the corrosion rates occur at about 900 F (see Appendix). The maximum effect of carbide precipitation on the mechanical properties, as might be expected, occurs at lower stress relief temperatures. The recovery of the room temperature transverse sharp-notch strength ratio at very high stress relief temperatures is due to agglomeration of carbides and further softening of the structure.

At −320 F the sharp-notch strength ratio varies with stress relief temperature in a manner somewhat different from that observed for room temperature tests. For both steels it is nearly constant to about 550 F. In the case of AISI 304 type L pronounced minima are observed at about 700 F. These are thought to be associated both with carbide precipitation and with the previously discussed slight elevation of the tensile strength at this temperature. At temperatures producing softening (above about 700 F) the sharp-notch strength ratio would be expected to increase with temperature. This behavior is observed for AISI type 304 L. However, in the case of AISI type 301 the ratios decrease continuously with increasing temperature above about 700 F, and the minima observed for room temperature tests are absent. This behavior does not imply that the effect of carbide precipitations are absent in the low temperature notch tests. Two effects act to depress the sharp-notch strength ratio of AISI type 301 in this temperature range. The embrittling effect of carbide precipitation tends to depress the notch strength. Furthermore, as discussed previously, the tensile strength is raised by the formation of martensite at low temperature. This further depresses the sharp-notch strength ratio and prevents its recovery at high stress relief temperatures.

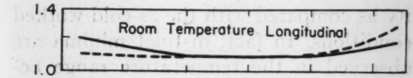


FIG. 15.—Sharp-Notch - Yield Strength Ratio at Room Temperature and −320 F for AISI Types 301 and 304 L Cold-Rolled 0.063-in. Sheet Stress Relieved at Various Temperatures.

The influence of stress relief may also be examined using the sharp notch yield strength ratio. This ratio has been plotted as a function of stress relief temperature in Fig. 15 for both AISI types 301 and 304 L.

For room temperature tests in the longitudinal direction the sharp-notch-yield strength ratio for both steels is above unity at all stress relief temperatures. This is also true at −320 F, except for AISI type 301 which exhibits a ratio of 0.9 at about 850 F.

Tests in the transverse direction are generally characterized by low ratios except at the highest stress relief temperatures, where appreciable softening occurs. Lower stress relief temperatures produced no decrease in notch sensitivity as compared with the as-cold-worked conditions. In fact, distinct minima are observed in the temperature range between 400 and 900 F. The presence of these minima indicates the lowest stress relief temperature should be used that produces the desired increase in yield strength.

SUMMARY OF RESULTS

The major contribution of this investigation lies in establishing the influence of very high stress concentrations on heavily cold-rolled and stress-relieved 18-8 type stainless steel sheet. Certain observations regarding the smooth tensile properties are also of interest since cold-reductions higher than generally studied were systematically investigated at both room temperature and −320 F.

The following behaviors of general interest appear reasonably well established by the data for both AISI type 301 and AISI type 304 L, 0.063 in. thick sheet. However, it should be remembered that the mechanical properties of these steels are strongly dependent on both the carbon and nickel content. Therefore, some variations in the magnitude of the observed effects may be noted within the normal limits of chemical composition. Furthermore, the effects of heat treatment are, of course, time-temperature dependent and the following statements apply only to the times investigated.

Effects of Cold Reduction:

1. In the as-rolled conditions, both steels exhibit negligible directionality of smooth tensile properties over the entire range of cold reductions investigated.
2. In contrast, pronounced directionality characterizes the sharp notch strength, with the transverse direction being considerably more notch sensitive.
3. Considering the sharp notch-yield strength ratio as an index of notch toughness, it may be concluded for both steels that a relatively high degree of toughness is maintained at −320 F in the longitudinal direction up to 70 per cent cold reduction. However, tests in the transverse direction reveal notch sensitivity which increases rapidly with the amount of cold reduction. For AISI type 301 steel, the transverse notch sensitivity is higher at −320 F than at room temperature, but for AISI type 304 L steel, the reverse in observed. For AISI type 301 the transverse notch sensitivity is higher at −320 F than at room temperature but for AISI 304 L alloy the reverse is observed. At 70 per cent cold-reduction, transverse sharp-notch-yield ratios less than 0.6 are obtained for AISI type 301 tested at room temperature or −320 F.

Effects of Stress Relief Heat Treatment:

1. The maximum elevation in yield strength for both steels tested at room temperature and −320 F, is developed by stress relief at 400 F for 24 hr.
2. Heating to temperatures higher than about 700 F generally produced loss in yield and tensile strength. However, for AISI type 304 L an elevation in the tensile strength occurs at about 700 F.
3. Stress relief at temperatures producing increases in the yield strength produced no reduction whatever in the notch sensitivity nor any decrease in the directionality of notch strength.
4. Heating in the temperature range between 600 and 900 F produced structural changes in both steels which adversely affected both the room tempera-

ture and −320 F sharp-notch properties, particularly in the transverse direction.

5. Boiling 65 per cent HNO_3 corrosion tests on highly cold-worked stress relieved steels showed generally low corrosion rates with the maximum rate being developed for stress relief temperatures about 900 F.

6. It would appear that the carbide precipitation responsible for the maximum in corrosion rate is also associated with the deterioration of the notch properties in the temperature range between 600 and 900 F.

7. For practical applications, stress relief should be carried out at as low a temperature as possible to avoid embrittlement.

Acknowledgments:

The authors are indebted to V. H. Leichliter, R. E. Griffith and C. H. Campbell of the American Steel and Wire Division of the U. S. Steel Corp. for furnishing the alloys. Particular appreciation is expressed to E. H. Phelps of the Applied Research Laboratory of the U. S. Steel Corp. for conducting the corrosion studies. Both George Sachs, Syracuse University, and M. H. Jones, National Aeronautics and Space Administration, were extremely helpful in discussions of the results.

J. L. Shannon Jr., and George Succop, National Aeronautics and Space Administration, assisted in the test program.

REFERENCES

(1) J. E. Srawley and C. D. Beachem, "Crack Propagation Tests of High Strength Steel Sheets Using Small Specimens," *NRL Rep. 5127*, Naval Research Laboratory, April 9, 1958.

(2) G. Sachs, "Survey of Low Alloy Aircraft Steel Heat Treated to High Strength Levels: Part 4.—High Strength Steels and Their General Static Properties," *TR 53-254*, Wright Air Development Center, Aug. 1954.

(3) G. B. Espey, M. H. Jones, and W. F. Brown, Jr., "A Preliminary Report on Sharp-Edge Notch and Smooth Tensile Characteristics of Ultra High Strength Steel Sheet Alloys," see p. 837, this publication.

(4) "Nickel Alloy Steels," Section 7, International Nickel Co., Inc., New York, N. Y. (1947).

(5) A. Pomp, A. Krisch, and G. Haupt, "Impact Strength of Alloy Steels at Temperatures of +20 to −253 C (Boiling Temperature of Hydrogen)," *Mitteilungen Kaiser Wilhelm Institut für Eisenforschung zu Düsseldorf*, Vol. 21, p. 219 (1939).

(6) V. N. Krivobok and A. M. Talbot, "Effect of Temperatures on the Mechanical Properties, Characteristics, and Processing of Austenitic Stainless Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, pp. 895–928 (1950).

(7) W. O. Binder, "Effect of Cold Work at Low Temperatures on Austenitic 18-8 Steel," *Metal Progress*, Vol. 58, Aug. 1950, pp. 201–207.

(8) N. A. Ziegler and P. H. Brace, "Hardening of Austenitic Stainless Steels by Mechanical Working at Subzero Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, pp. 861–879 (1950).

(9) R. Franks and W. O. Binder, "Effects of Low Temperature Heat Treatment on Elastic Properties of Cold-Rolled Austenitic Stainless Steels," *Transactions*, Am. Inst. Mining and Metallurgical Engrs. (Iron and Steel Div.), Vol. 140, pp. 433–448 (1940).

(10) R. Franks and W. O. Binder, "Tension and Compression Stress Strain Characteristics of Cold-Rolled Austenitic Chromium-Nickel and Chromium-Manganese-Nickel Stainless Steels," *Journal of the Aeronautical Sciences*, Vol. 9, p. 419 (1942).

(11) D. J. McAdam and R. W. Mebs, "Tensile Elastic Properties of 18:8 Chromium-Nickel Steel as Affected by Plastic Deformation," *NACA Report 670* (1939).

(12) D. J. McAdam and R. W. Mebs, "Tensile Elastic Properties of Typical Stainless Steels and Nonferrous Metals as Affected by Plastic Deformation and by Heat Treatment," *NACA Report 696* (1940).

(13) E. M. Mahla and N. A. Nielsen, "Carbide

Precipitation in Type 304 Stainless Steel—An Electron Microscope Study," *Transactions,* Am. Soc. Metals, Vol. 43, pp. 290–314 (1951).

(14) E. H. Schmidt, "Low Temperature Impact of Annealed and Sensitized 18-8 Steel," *Metal Progress,* Vol. 54, p. 698 (1948).

(15) V. N. Krivobok, "Properties of Austenitic Stainless Steels at Low Temperatures," *Circular 520,* Nat. Bureau of Standards (1952).

(16) A. Kramer and W. M. Baldwin, Jr., "Carbide Precipitation and Brittleness in Austenitic Stainless Steel," *Transactions,* Am. Soc. Metals, Vol. 50, pp. 803–812 (1958).

APPENDIX I

CORROSION TESTS

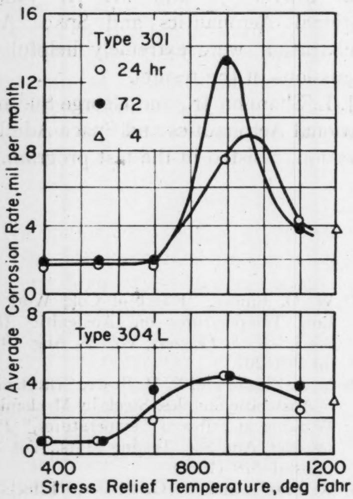


FIG. 16.—Average Corrosion Rate in 65 per Cent Boiling Nitric Acid for Five 48-hr Periods (ASTM Recommended Practice A 262 – 55 T) as a Function of Stress Relief Temperature for AISI Types 301 and 304 L 0.063-in. Cold-Rolled Sheet.

Sheared 1½ by 2½ in. sample blanks of both AISI type 301 (60 per cent cold-rolled) and AISI 304 type I (70 per cent cold-rolled)[5] were subjected to the same stress relief heat treatments used in the study of the mechanical properties. In addition, a set of samples were given a 4-hr 1200 F heat treatment which is known to produce "sensitization" in annealed AISI type 304.

These blanks were then milled to a 1 by 2 in. corrosion specimen and polished on the edges and surfaces with 120 grit paper. The specimens were then scrubbed and degreased in acetone, treated in 20 per cent HNO_3 at 120 and 140 F for 20 min and then rinsed in distilled water prior to initial weighing. The corrosion tests in 65 per cent boiling HNO_3 were carried out in accordance with ASTM Recommended Practice A 262 – 55 T.[6] Single specimens were tested in each flask.

The results are given in Fig. 16 which shows the average corrosion rate (average of five 48-hr test periods) as a function of the stress relief temperature. As might be expected the lowest rates are observed for the extra low carbon AISI type 304. Both steels exhibit a maximum at about 900 F which is generally associated with carbide precipitation. For AISI type 301 a small effect of stress relief time appears, with the maximum rate being shifted to lower temperatures for the longer heat-treatment time. This might be expected because the carbide precipitation is a time-temperature-dependent phenomenon. For AISI type 304 L the corrosion rates are so low that the scatter masks the time effect.

---

[5] The preparation of the specimens and corrosion testing were done at the Applied Research Laboratory, U. S. Steel Corp.

[6] Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 – 55 T), 1958 Book of ASTM Standards, Part 3, p. 288.

DISCUSSION

Mr. E. J. Dulis.[1]—Although the steels investigated are austenitic in the annealed condition, they are quite unstable and transform to martensite during cold working. Therefore, did the authors measure the amount of martensite that formed and attempt to relate this quantity with the characteristics of the steels, after the cold working? Also, have the authors considered testing a stably austenitic steel—that is, one that does not form martensite during cold working, after different amounts of cold reduction?

Mr. D. W. McDowell, Jr.[2] (*by letter*).—The authors should be congratulated on this contribution to our knowledge on the effect of cold working plus post-rolling annealing of the austenitic stainless steels. Since the original work done by Krivobok and Talbot (references (6,15) of the paper), International Nickel Co.'s Bayonne Research Laboratory has investigated other austenitic stainless steels. For example, it was found that, by subzero rolling and post-rolling heat treatments, strength improvement was greater for type 347, columbium stabilized stainless steel, than for types 301 and 302 steels. Upon further investigation in which changes to the nickel and columbium levels were examined, it was found that properties in excess of 300,000 psi tensile strengths could be obtained by these steels.

It has already been shown that certain compositions in the iron-chromium-nickel system, although predominantly austenitic at room temperature, yielded an austenite which was sufficiently metastable to undergo partial transformation to ferrite upon exposure to subzero temperatures. As discussed by Krivobok and Talbot, this metastable austenite field includes many of the commercially significant types 300 series stainless steels. The essential findings of the work cited in this early work were threefold. First, it was demonstrated that low temperature mechanical working effected substantially larger changes in mechanical properties than purely thermal treatment at low temperatures. Strength and hardness increased with a decrease in working temperature and an increase in the amount of working at temperature, although the rate of change of properties with respect to the temperature and working variables decreased sharply, for some alloys, beyond certain values of these variables. With respect to room temperature rolled sheet of the same composition, tensile strength increases of the order of 100,000 psi were gained through the agency of sub-zero working. Second, it was shown that an annealing treatment subsequent to the sub-zero rolling (hereinafter abbreviated to "zerolling" after Messrs. Samans and Shortsleeve) yielded an additional strength advance, in some instances amounting to 100,000 psi. A maximum annealing effect was found for temperatures and times ranging from 500 to 900 F and 1 to 500 hr respectively. With temperatures in excess of roughly 100 F, the annealing effect was found to deteriorate sharply. Third, it was shown that the spectacular strength augmentation imparted to certain alloys by zerolling plus annealing was retained up to at least 800 F and that tensile properties were insensitive to a holding time at temperature of at least 100 hr.

---

[1] Research Supervisor, Crucible Steel Company of America, Pittsburgh, Pa.

[2] International Nickel Co., New York, N. Y.

The effects of zerolling plus annealing appear to be primarily dependent on two factors, namely, the stability of the retained austenite in the unworked metal and the strain aging characteristics of the duplex, ferritic-austenitic, structure resulting from the zerolling treatment. A number of papers concerning the gamma to alpha transformation in the iron-nickel-chromium systems have appeared in recent years. This paper by Espey, Repko, and Brown is one of the better of these.

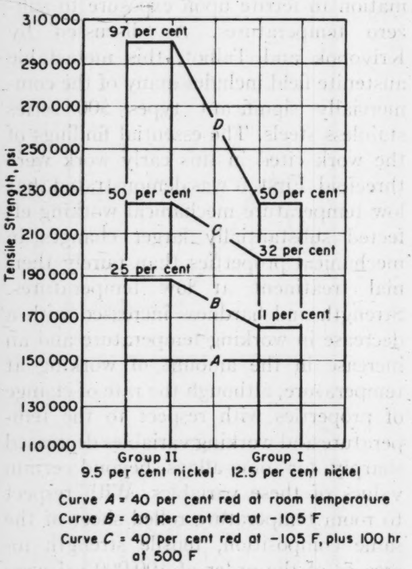


FIG. 17.—Strength Increases in Group I and Group II Steels Effected by Various Treatments.

One comment should be made concerning these authors' paper: no attempt was made to measure crack length. This critical crack length is of much interest to the missile industry and could be determined by a marking fluid such as India ink.

Although a large number of stainless steels were tested at the Bayonne laboratory of the International Nickel Co., the program was narrowed down to examine the response of type 347 steel in somewhat greater detail, particularly with reference to changes in the nickel and columbium levels. Figure 17 shows the strength increases in group I and group II steels effected by various treatments.

Group I steels have 0.05 carbon, 12.5 nickel, 18.2 chromium, 0.98 columbium, 0.43 aluminum, 0.57 silicon and 1.15 manganese.

Group II steels contain 0.06 carbon, 9.5 nickel, 18.4 chromium, 0.95 columbium, 0.42 aluminum, 0.65 silicon and 1.29 manganese.

To summarize the results of this laboratory investigation, the following conclusions can be made:

1. Tensile and yield strengths of 300,000 and 290,000 psi respectively, have been exhibited by steels of 18.5 chromium, 9.5 nickel, containing columbium (0.9 to 1.1 per cent) and aluminum (0.3 to 0.8 per cent). These properties were attained through the agency of a 40 per cent reduction at −105 F and a post-rolling anneal of 100 hr at 800 F.

2. Relative to conventionally rolled material, the absolute strength gain derived from identical rolling plus annealing was about 100 per cent greater for the 18.5 chromium, 9.5 nickel-base steels than for the 18.5 chromium, 12.5 nickel steels. X-ray determinations of austenite showed austenite contents of about 35 per cent and 93 per cent, for the 9.5 and 12.5 nickel steels, respectively, after the sub-zero rolling plus annealing treatment. With respect to the response to this treatment, it is evident that a closer

specification of nickel content in the 18-8 grade stainless steels is mandatory.

3. The substantial strength improvements, relative to conventionally rolled sheet, imparted to the 18.5 chromium, 9.5 nickel-base steels by sub-zero rolling plus annealing is retained up to temperatures in the order of 900 F. At 800 F, a tensile strength and elongation of 220,000 psi and 5 per cent respectively were registered by the 18.5 chromium, 9.5 nickel steel with either columbium or aluminum. Above about 800 F, the onset of a rapid deterioration of strength with temperature increase was observed.

4. It was found that the properties of the sub-zero rolled and annealed steels could be correlated with a Larsen-Miller type heat-treat parameter. With respect to this parameter, a sharp maximum in strength was observed. It is believed that this point of maximum annealing effect is related to the reestablishment of austenite.

5. The property response of the 18 chromium, 9.5 nickel-base steels to the sub-zero rolling and annealing was practically unaffected by the following variation in alloy content: 0.9 per cent columbium plus aluminum ranging from 0.3 to 0.7; 1.1 per cent columbium and no aluminum; 0.8 per cent aluminum and no columbium.

To supplement the above investigation samples of the aluminum-modified type 347 stainless steel (9.5 per cent nickel) were tested at Southern Research Institute at a nominal strain rate of 0.1 in. per in. per sec.

TABLE II.—TENSILE PROPERTIES OF 347 STAINLESS STEEL SHEET[a] AT DIFFERENT TEMPERATURES, AND AT A NOMINAL STRAIN RATE OF 0.1 IN. PER IN. PER SEC.
All specimens heated to test temperature within 20 sec and held for 10 sec

Test Temperature, deg Fahr	Strain Rate, in. per in. per sec	Stress in 1000 psi at					Modulus of Elasticity, 10^6 psi	Total Elongation in 2 in., per cent
		0.2 per cent Offset	0.5 per cent Strain	1.0 per cent Strain	2.0 per cent Strain	Ultimate Stress		
75	0.395	329.0	[b]	[b]	[b]	329.0	26.7	1.0
	0.35	334.0	[b]	[b]	[b]	[c]	26.5	[c]
800	0.324	229.0	[b]	[b]	[b]	229.0	23.2	1.0
	0.33	225.5	[b]	[b]	[b]	225.5	22.0	1.0
1000	0.233	140.5	149.8	145.0	[b]	149.8	25.1	2.5
	0.316	154.5	157.5	149.8	[b]	157.5	21.4	2.0
1200	0.25	88.0	98.7	105.53	105.3	106.3	17.3	4.5
	0.291	89.2	101.0	106.5	104.2	106.5	18.2	5.5

[a] 40 per cent cold-reduced at −105 F and stress-relief annealed at 800 F for 24 hr. The prereduction thickness was 0.060 in.

[b] Specimen did not reach indicated strain.

[c] Specimen broke outside gage section.

Table II shows the results of these tests as the temperature was raised from room temperature to 1200 F. The strength properties drop sharply as the test temperature was raised; however, room temperature strength properties as high as 329,000 psi were obtained.

At the present time Southern Research Institute is testing austenitic stainless steels rolled (from an original 0.060 in. thickness) with up to 80 per cent reduction at −105 F and with up to 40 per cent reduction at −320 F. Results of these tests will be forthcoming soon.

MESSRS. G. ESPEY, A. J. REPKO AND W. F. BROWN, JR. (*authors' closure*).—In answer to Mr. Dulis, no determinations of martensite were made. This is because no simple method has yet been devised. Very steep plastic strain gradients exist at the notch. It is this very small region which transforms and controls the fracture process. Magnetic techniques might be overly insensitive or misleading. Stable austenite has not been tested, as yet, unless the low carbon type 304L may be considered as approaching such a steel. It may be any martensite formed in this steel is appreciably closer to the properties of the remaining cold worked austenitic matrix than is the case for the high carbon, higher martensite stainlesses. Some testing on fully stable 310 will probably be conducted in the future. Indeed, more quantitative information is needed on the effect different amounts of martensite may have on the sharp notch properties of different compositions.

Mr. McDowell, Jr., presents some very interesting data which should certainly stimulate further work on sub-zero strengthening. It is to be noted that relatively small reductions might be expected to produce large increases in strength if the stainless steel is sufficiently unstable (that is, low in nickel content). One might visualize the use of such steels where the fabrication of the component could be carried out at low temperature. For example, this might mean final sizing of a missile case by hydrostatic pressure at low temperature. Generally, the properties of work-strengthened metastable austenite in either stainless or low-alloy steels are of great interest to the missile designer and others. Mr. McDowell mentions the use of ink staining to follow slow crack development. This method is now used by the authors on all notch specimens tested at room temperature. At the time of this investigation, the technique was not known to us.

THE SHARP EDGE NOTCH TENSILE STRENGTH OF SEVERAL HIGH-STRENGTH STEEL SHEET ALLOYS*

BY G. B. ESPEY,[1] M. H. JONES,[1] AND W. F. BROWN, JR.[1]

SYNOPSIS

A sharp edge notch tension test was used to evaluate the room temperature notch sensitivity of several high-strength sheet steels. These steels are being considered for application in light weight highly stressed pressure vessels. The notch test was designed primarily to permit rapid evaluation of an alloy's tendency to fail at low stresses in the presence of high stress concentrations. The test qualitatively simulates the embrittling action of defects such as cracks or areas of nil ductility. It is believed to be sufficiently severe in this respect to give meaningful results when applied to the present light weight pressure vessel problem.

Sharp edge notch and smooth tensile characteristics are reported for representative steels from the following classes: (1) ferritic low alloy steels; (2) hot-worked tool steels; (3) martensitic stainless steels; (4) precipitation hardening stainless steels; and (5) cold rolled stainless steels. Particular emphasis was given to revealing embrittlement ranges associated with heat treatment or cold work producing a wide range of strength levels.

The data reveal the extreme sensitivity of the sharp edge notch strength to known embrittling heat treatments and exhibit excellent reproducibility. It is definitely established that the smooth tensile properties give no direct indication of notch sensitivity. Fully notch ductile behavior was not obtained with this test for tempered martensites at tensile strength levels over 200,000 psi. Therefore, basing a component design primarily on the tensile yield strength may be a very dangerous procedure. At strength levels above 200,000 psi notch sensitivity varies considerably depending on the alloy, with the newer ultra-high-strength materials exhibiting considerable embrittlement.

The use of heat-treated alloy steels at very high strength levels frequently has resulted in brittle fracture at average stresses only a fraction of the yield strength. Several examples of such failures have been reviewed by Sachs (1).[2] The problem has recently again been brought into sharp focus by the application of ultra-high-strength sheet alloys in certain welded large solid propellant missile casings. The application is at room temperature or somewhat above. The need for weight savings is even more critical than in aircraft, and the desired strength levels are higher than ever before employed in sheet alloys.

Previous experience with brittle failure of high-strength steel components has shown that such fractures are always associated with the presence of known embrittling factors. These include stress concentrations, low temperatures, high loading rates, hydrogen embrittlement, and large size. Outstanding is the influence of stress concentrations. These are not only associated with discontinuities such as threads or fillets but also arise from internal "flaws." Such flaws may be small cracks resulting from welding or heat treatment, inclusions, or areas of exceptionally low ductility. It has been shown repeatedly (2,3,4) that the size of a critical flaw decreases with an increase in the strength level of the steel.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] National Aeronautics and Space Administration, Lewis Research Center, Cleveland, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Unfortunately, there does not appear to be a general awareness of the above described facts. Thus plans and designs for application of high-strength steels are still often based on the yield strength alone. Furthermore, the course of high-strength alloy steel development has been affected largely through conventional (smooth) tensile data supplemented by standard impact tests. Smooth tensile data gives no indication of possible weakness in the presence of stress concentrations. While the impact test contains a stress concentration, frequently it is not sufficiently severe to reveal embrittlement which may occur in service. As has been emphasized in this country by Sachs (2), Irwin (5,6), and others (7,8) and in Germany by Buchmann (9), Kuntze (10), Richard (11), and others, any meaningful material evaluation must reveal the effects of high stress concentrations. This may be achieved in the laboratory by tests at the service temperature with specimens containing machined sharp notches (12) or induced cracks (6,8). Another approach, used by Puzak and Stoop (13), focuses attention on the fracture appearance transition temperature in simple impact-die bulge tests on sheets containing stress concentrations.

In the authors' opinion, the machined sharp notch tension test as first used by Sachs and Brown (12,14) in the evaluation of heat-treated low-alloy steels offers the simplest and most direct procedure for determination of the effect of very high stress concentrations. For steels heat treated to high strength it provides, in conjunction with the smooth tension test, a direct indication of an alloys' ability to eliminate high stress concentrations by plastic flow.

A considerable amount of data exists regarding the strength of circumferentially sharp-notched cylindrical specimens of several common heat-treated low-alloy constructional steels. These data have recently been reviewed by Sachs (16) and definitely show that severe embrittlement is characteristic of certain compositions at high strength levels. However, essentially no similar information exists in the published literature for sheet alloys and very little notch tensile data are available for the newer "high-bred"[3] steels and the effects of melting practices on these steels.

The object of the present investigation was to evaluate rapidly the relative resistance to high stress concentrations of several ferritic and austenitic sheet alloys of interest for use in thin-walled pressure vessels. As will be discussed later, the test geometry selected for this purpose represents a rather severe flaw in any reasonably designed pressure vessel. Therefore, the test appears to be sufficiently sensitive to yield meaningful qualitative results for a wide variety of high strength steels. Particular emphasis has been placed on revealing any ranges of embrittlement associated with heat treatments producing tempered martensites or conditions of cold work that produce a wide range of strength levels. The

---

[3] These steels are more highly alloyed than SAE 4340 steel. They were developed to possess high hardenability, high impact strength, and superior properties at high strength levels.

TABLE I.—CHEMICAL COMPOSITION OF SHEET ALLOYS.

Alloy	Thickness, in.	C melt	C sheet^c		Composition, per cent											Supplier	Heat, number
			As received	As heat-treated	Manganese	Phosphorus	Sulfur	Silicon	Nickel	Chromium	Vanadium	Molybdenum	Aluminum	Nitrogen	Copper		
SAE 4340^b	0.095	0.40	0.37	0.35	0.69	0.010	0.012	0.29	1.77	0.74	...	0.23	0.01	...	...	Republic Steel Corp.	H58807
AMS 6434^b	0.095	0.36	0.36	0.34	0.74	0.008	0.008	0.28	1.72	0.76	0.20	0.33	0.03	...	...	Republic Steel Corp.	...
AMS 6434^b	0.063	0.344	0.35	0.35	0.75	0.008	0.004	0.40	1.78	0.86	0.18	0.28	...	...	...	Allegheny-Ludlum Steel Corp.	22138
AMS 6434 CEVM^{a,b}	0.063	0.342	0.32	0.32	0.70	0.009	0.004	0.40	1.76	0.90	0.18	0.30	...	...	...	Allegheny-Ludlum Steel Corp.	04104
UHS 280^b	0.063	0.41	0.40	0.38	1.20	0.014	0.020	1.10	...	1.20	0.16	0.33	...	...	...	Crucible Steel Co. of Am.	71396
300 M^b	0.063	0.41	0.40	0.38	0.85	0.012	0.005	1.75	1.88	0.78	0.14	0.31	...	...	...	Ingersoll Steel Co.	3266
X 200	0.063	0.43	0.44	0.43	0.89	0.013	0.015	1.53	0.10	2.05	0.07	0.52	...	...	0.09	U. S. Steel Corp.	X38832
Vascojet 1000^b	0.063	0.43	0.43	0.43	0.31	0.017	0.011	0.93	...	5.24	0.55	1.28	...	...	...	Vanadium-Alloys Steel Corp.	30625
AISI 410	0.063	0.11	0.11	0.11	0.54	0.016	0.014	0.26	0.10	12.25	...	...	...	...	...	Crucible Steel Co. of Am.	122396
12 MoV	0.063	0.23	0.23	0.23	0.50	0.020	0.013	0.50	0.79	12.40	0.31	0.96	...	...	...	U. S. Steel Corp.	X19787
AM 350	0.063	0.089	...	...	0.76	0.018	0.017	0.24	4.11	16.37	...	2.73	...	0.090	...	Allegheny-Ludlum Steel Corp.	79738
17-7 PH	0.063	0.065	...	...	0.69	0.020	0.009	0.28	7.11	16.96	...	...	1.13	...	...	Armco Steel Corp.	66875
PH 15-7 Mo	0.063	0.070	...	...	0.66	...	...	0.47	7.20	15.08	...	2.40	1.16	...	...	Armco Steel Corp.	880362
AISI 304 L	0.063	0.017	...	...	1.38	0.016	0.019	0.65	9.44	18.18	...	...	...	...	...	U. S. Steel Corp.	7X1964
AISI 301	0.063	0.10	...	...	1.24	0.033	0.020	0.53	7.28	17.16	...	...	...	...	...	U. S. Steel Corp.	9X9343

[a] Consumable electrode vacuum melt.

[b] Cross-rolled from sheet bar.

[c] At one quarter width position, determined by American Car and Foundry Division, ACF Industries, Berwick, Pa.

influence of sheet directionality is also established. In addition, preliminary results are presented for effects of vacuum melting. The data are analyzed to reveal the relative embrittlement characteristic of the various alloys at presently used and projected strength levels. A comparison is made with published data related to specimens having induced actual cracks. Furthermore, from the present data and that previously published for circumferentially notched bars it is possible to arrive at certain generalizations of interest for the fabricator, designer, and alloy producer.

TABLE II.—SUPPLIERS INCLUSION RATINGS (ASTM RECOMMENDED PRACTICE E 45 – 51, SECTION A) FOR SHEET ALLOYS.[a]

Alloy	Type A		Type B		Type C		Type D	
	Thin	Heavy	Thin	Heavy	Thin	Heavy	Thin	Heavy
AMS 6434, 0.063-in. air melt, sheet	0	0	3	0	0	0	2.5	0
AMS 6434, 0.063-in. CEVM, sheet	0	0	1	0	1	0	1.5	0
AMS 6434, 0.063-in. CEVM, sheet, bar top center	1	0	0	0	0	0	1.5	0
AMS 6434. 0.063-in. CEVM, sheet bar, bottom center	1.5	0	0	0	0	0	1	0
UHS 280, 0.063-in., sheet	¼	0	2½	0	0	0	0	2
300 M, 0.063-in., sheet	0	0	0	0	1.5	1.5	0	0
X-200, 0.063-in., sheet	0	0	0	0	2	0	0	0
Vascojet 1000, sheet	0	0	−1	0	0	0	2	0
AISI 410, sheet	1	0	2	0	0	0	1	0
12 MoV, sheet edge	3	0	0	0	4	0	0	0
12 MoV, sheet center	4	0	0	0	4	0	0	0
AM 350, sheet	0	0	2	0	3	0	2	0
PH 15-7 Mo, sheet	0	0	3	0	0	0	0	0
AISI 304L, sheet edge	3	0	0	0	4	0	0	0
AISI 304 L, sheet center	4	0	0	0	4	0	0	0
AISI 301, sheet edge	3	0	0	0	4	0	0	0
AISI 301, sheet center	3	0	0	0	3	0	0	0

[a] Sheet ratings at one quarter width position unless indicated.

MATERIALS

The compositions of the alloys thus far evaluated are listed in Table I. Unless otherwise noted all alloys were cross rolled from the sheet bar. With the exception of cold-rolled AISI types 304L and 301 steels, all sheet was received in the hot-rolled and annealed condition. The standard thickness for this phase of the investigation was a nominal $\frac{1}{16}$ in. With few exceptions, the alloys were available in this gage.

In an attempt to determine the presence or absence of decarburization, carbon analyses[4] were made on the as-received and as-heat-treated sheet at the one-quarter width position. The carbon analysis information was supplemented by metallographic studies in cases where carbon loss occurred.

It has been suggested that inclusions are particularly damaging to the transverse properties and also exert a deleterious effect on the longitudinal notch strength. For this reason an attempt was made to obtain inclusion ratings according to ASTM Recommended Practice E 45 – 51 (method A)[5] for all the alloys.

---

[4] The authors are indebted to W. Ruprecht of the American Car and Foundry Division, ACF Industries, Berwick, Pa. for furnishing the sheet carbon analysis.

[5] Recommended Practice for Determining the Inclusion Content of Steel (E 45 – 51), 1958 Book of ASTM Standards, Part 3, p. 529.

However, such information is not normally furnished for sheet products. Therefore, in most cases the conventional billet ratings were not available, and inclusions counts were made on the sheets, again at the one quarter width position. These findings are shown in Table II.

PROCEDURE

Specimen blanks 2 by 8 in. were sheared from the as-received sheet stock and the specimens machined from these blanks. Machining was confined to the sheet edges, since pressure vessels are not normally subjected to any surface machining.

Heat treatment, in all cases, was carried out after finish machining and degreasing. Again, this approximates the actual conditions encountered in fabrication of pressure vessels. In nearly all vessels the welding is carried out prior to heat treatment. Thus, surface cracks resulting from either welding or heat treatment are subject to any possible chemical action of the heat treating or stress relieving procedure. The individual heat treating schedules are shown in Table III. As indicated, all ferritic alloys were austenitized in argon and oil quenched, with the exception of Vascojet 1000. Vascojet 1000 was austenitized in a sealed Inconel tank which had been previously evacuated and charged with argon ($\frac{1}{4}$ atmos pressure). The sealed tank was cooled in air, the cooling rate being approximately 100 F per min between 1850 and 1000 F. The precipitation hardening austenitic alloys were sensitized in air. All tempering (or aging) was in air. Every attempt was made to follow the alloy suppliers' recommendations regarding austenitizing (or sensitizing) temperature and time and the tempering times. Hardness readings were determined after austenitizing (or sensitizing) on selected specimens and on every specimen following tempering.

Specimen Selection:

The selection of a suitable smooth specimen (Fig. 1) presents no particular problem. It was necessary only to so proportion the dimensions that fracture occurs in the gage section for the most brittle conditions.

As emphasized by Irwin (5,6), if service failures are occurring at stresses below the yield strength a meaningful laboratory notch test must also produce failures at these low strength values. In some ductile alloys, this requires the use of very large specimens. However, for the evaluation of high-strength steels relatively small sizes should give pertinent information providing the initial stress concentration is sufficiently high.

TABLE III.—HEAT TREATMENT SCHEDULE FOR THE SHEET ALLOYS.[a]

SAE 4340	1520 F, ½ hr; oil: 2 hr temper
AMS 6434	1620 F, ½ hr; oil: 2 hr temper
UHS 280	1725 F, 10 min; oil: 1 hr temper
300 M	1700 F, ½ hr;[b] 1600 F, ½ hr, oil: 1 hr temper
X-200	1750 F, ¼ hr; oil: 1 hr temper
Vascojet 1000	1850 F, ½ hr, air; triple temper 3 hr total
AISI 410	1800 F, ½ hr, oil; 3 hr temper
12 MoV	1850 F, ¼ hr, oil; 4 hr temper
AM 350	1710 F (air), 10 min; air: −110 F, 3 hr minimum: 3 hr temper
17-7 PH and PH 15-7 Mo	1750 F (air), 10 min; air: −110 F, 8 hr minimum: 1 hr temper

[a] All high-temperature treatments in argon except where noted and all tempering in air.

[b] Furnace temperature reduced from 1700 to 1600 F.

The results of Sachs et al (15) permit the design of a notch specimen particularly suitable for testing sheet alloys.

These results relate to the influence of thickness to width ratio, notch depth, and notch radius on the room temperature notch strength of SAE 4340 rectangular specimens heat treated to high strength level.

On the basis of these data, the edge notch sheet specimen selected for this investigation (Fig. 1) had an over-all width of 1 in. (width to thickness ratio equal to 16) and a notch depth of 30 per cent. The 60-deg V-notch was provided with a root radius of less than 0.001 in. The specimen qualitatively simulates the effect of a crack 0.3 in. long in a pressure vessel. This is considered to be a size readily detectable by nondestructive inspection techniques and probably a more severe condition than would be encountered during reasonably good welding practice.

It is considered important that the notch radius be kept as small as possible. Thus, the tests on rectangular specimens (15) show a continuously decreasing notch strength ratio for radius values down to 0.001 in. Notch radii less than 0.001 in. are readily machined into materials with hardnesses up to about Rockwell hardness C 50 using the techniques outlined in Appendix I. The radius produced is difficult to resolve at 100× magnification. The actual values of radii may vary from specimen to specimen. However, the absence of excessive scatter even for the extremely brittle conditions indicates that either the radii are below a critical value where further decrease would produce no further embrittlement or the radii actually produced are quite uniform.

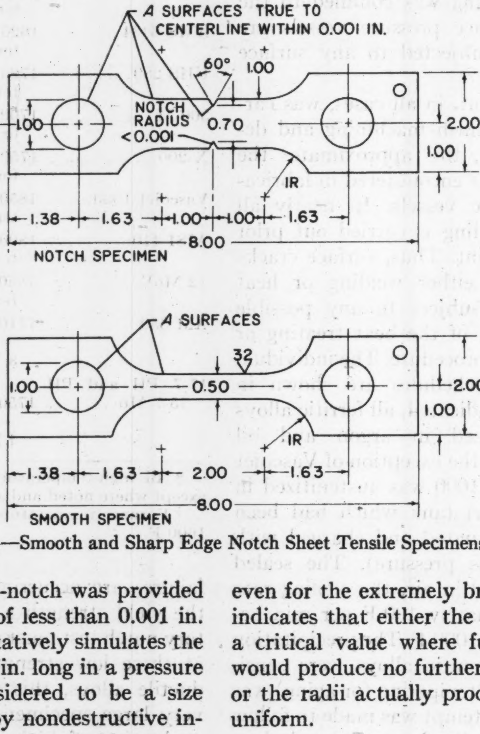


FIG. 1.—Smooth and Sharp Edge Notch Sheet Tensile Specimens.

Testing Techniques:

The requirements for low eccentricity of loading when testing brittle materials have already been discussed in detail (18). In the present investigation the specimens were pin loaded using a finish ground Kennametal pin held in an accurately machined yoke. Bending moments perpendicular to the sheet plane are of no great consequence because of the relatively thin stock employed. Bending moments in the sheet plane can arise either because of misalignments characteristic of the specimen itself or due to external eccentricities of load application. The former effects were minimized by very careful location of the pin loading holes and notches in relation to the loading axis. Eccentricities due to misalignments external to the specimen are compensated in this case by rotation of the specimen about the pin. This can take place at low loads because of low friction between the pins and the smooth surfaces of the specimen pin holes.

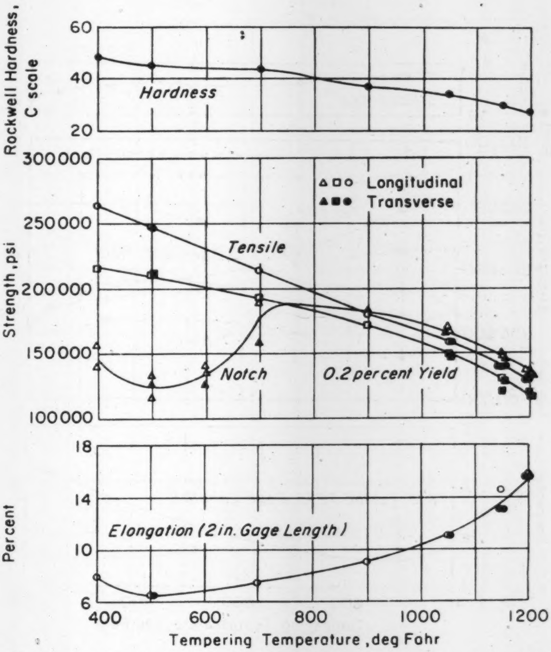


FIG. 2.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for SAE 4340 Air Melt 0.095-in. Sheet as a Function of Tempering Temperature.

The effectiveness of the above procedures in reducing the eccentricity is indicated by the good agreement in notch strength between replicate tests on the very brittle conditions. The scatter is generally no greater than previously reported for the strength of circumferentially notched buttonhead cylindrical specimens of brittle low-alloy steels tested in specially designed concentric fixtures (**12,14**).

The strain in a 2-in. gage length was measured as a function of load using an O.S. Peters extensometer in connection with a Templin type recorder. Autographic curves generally extending to only a few per cent strain were used to obtain the 0.2 per cent yield strength. Elongations are reported for a 2-in. gage length and were determined using light scribe marks on the specimen surface. The maximum loading rate corresponded to a free running head movement of 0.040 in per min.

Results and Discussion

For the purposes of this paper, the alloys may be divided into three general classes, namely, (1) ferritic steels; (2) precipitation hardening stainless steels, AM 350, 17-7PH, and PH15-7Mo; and (3) the cold-worked austenitic stainless steels AISI types 304L and 301. The ferritic steels may be subdivided into: (1) low-alloy steels, SAE 4340, AMS 6434, UHS 280, 300 M and X200; (2) hot-work tool steels, Vascojet 1000; and (3) martensitic stainless steels AISI types 410 and 12 MoV. The results will be presented for each general class separately.

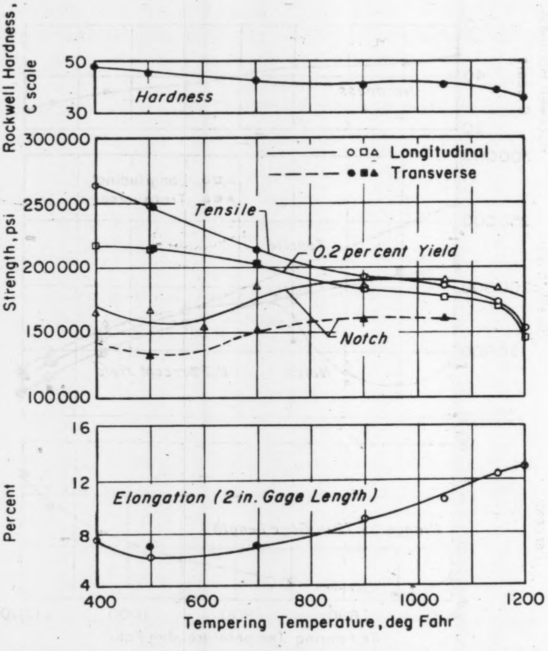


Fig. 3.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for AMS 6434 Air Melt 0.095-in. Sheet as a Function of Tempering Temperature.

The data are presented in Figs. 2 to 16 as a function of the primary variable controlling the strength level, namely, the tempering temperature or the amount of cold reduction. These representations reveal the influence of the primary variables on the smooth tensile characteristics. The notch strength[6] trend clearly defines regions of embrittlement.

In the following discussion, particular emphasis will be placed on the heat treatment and compositional variables influencing the notch sensitivity. For purposes of the present discussion a notch-sensitive alloy condition is one characterized by a sharp notch strength less than the tensile strength, as will be discussed later.

---

[6] The notch strength and tensile strength are nominal values determined by dividing the maximum load by the original minimum areas.

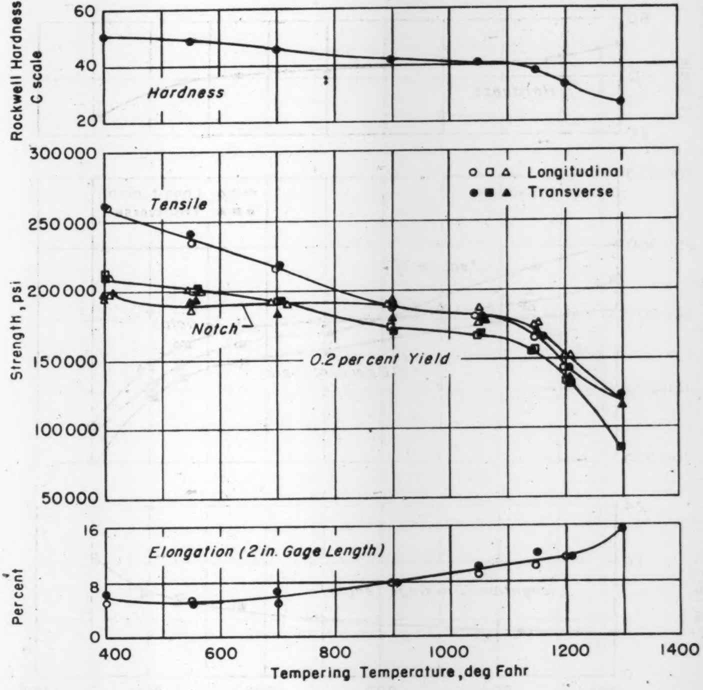


FIG. 4.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for AMS 6434 Air Melt 0.063-in. Sheet as a Function of Tempering Temperature.

Smooth Properties of the Ferritic Steels:

These properties have been sufficiently well investigated (19) so that certain fundamental behaviors are established and these are reflected in the present results. These behaviors will be briefly reviewed since the metallurgical phenomena responsible for them may also influence the notch strength.

Regarding the tensile and yield strengths, the basic behavior is typified by SAE 4340 steel (Fig. 2) which exhibits a continuous decrease in tensile strength with increasing tempering temperature. The ratio of yield strength to tensile strength for this steel first increases with an increase in tempering temperature to a maximum value (0.95) between about 800 to 900 F and then decreases slightly at higher tempering temperatures. The low yield ratio at low tempering temperatures is caused by the retention of residual quenching stresses of the first and second order which depress the stress-strain curve at small plastic strains.

This basic behavior is modified by the

addition of various carbide-forming alloy elements which result in secondary hardening and a consequent increased resistance to softening at the higher tempering temperatures. These additions result in the appearance of "humps" in the tensile and yield strength curves of the higher alloyed materials (Figs. 3 to 11). The clearest example of the effect of secondary hardening is offered by the hot-work tool steel Vascojet 1000 (Fig. 9) and the 12-chromium stainless steels (Figs. 10 and 11). These alloys exhibit a tensile strength at high tempering temperatures equal to or greater than at low tempering temperatures. The lower alloyed materials AMS 6434, UHS 280, 300 M and X-200 (Figs. 3 to 8) also exhibit the effects of secondary hardening but to a lesser degree. The combination of residual stresses and secondary hardening produce quite complex trends in the yield strength. Thus, with decreasing tempering temperature, the yield strength may be essentially constant or even decreasing while the tensile strength is increasing (for example, UHS 280 alloy, 600 to 400 F; 300 M alloy, 700 to 400 F; X-200 alloy, 700 to 400 F).

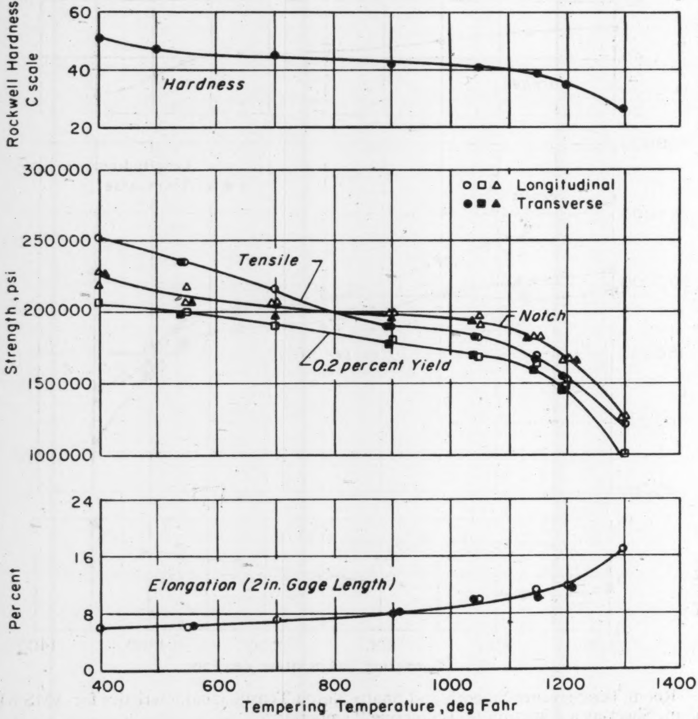


FIG. 5.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for AMS 6434 Consumable Electrode Vacuum Melted 0.063-in. Sheet as a Function of Tempering Temperature.

It is of interest to compare the as-quenched hardness of the ferritic alloys with values which might be expected on the basis of heat-treated sheet carbon content. According to Fig. 12 the hardness deviates downward from the ideal curve (100 per cent martensite) (30) for several of the sheet alloys. Some small departure from the ideal behavior is known to exist for nearly all high-strength low-alloy steels (19) and is presumably explained by the retention of austenite. However, for both SAE 4340 and AMS 6434 steels the deviation is greater than might be expected. Furthermore, it will be noted from Figs. 2 to 5 that the tensile and yield strengths of these alloys are lower than might be expected on the basis of their carbon contents. Thus, examination of bar stock tensile data (31) for oil-quenched SAE 4330 steel (0.30 carbon) indicates the yield strength of this bar stock, at tempering temperatures less than about 700 F, is from 5000 to 10,000 psi higher than observed for SAE 4340 and AMS 6434 0.095-in. sheet steel. Furthermore, the yield strengths of the bar stock are 15,000 to 20,000 psi higher than the corresponding values for 0.063-in. AMS 6434 sheet steel. Similar but much smaller differences are noted for the tensile strengths. These results are rather surprising since the bar stock contained 4 points lower carbon than the sheet

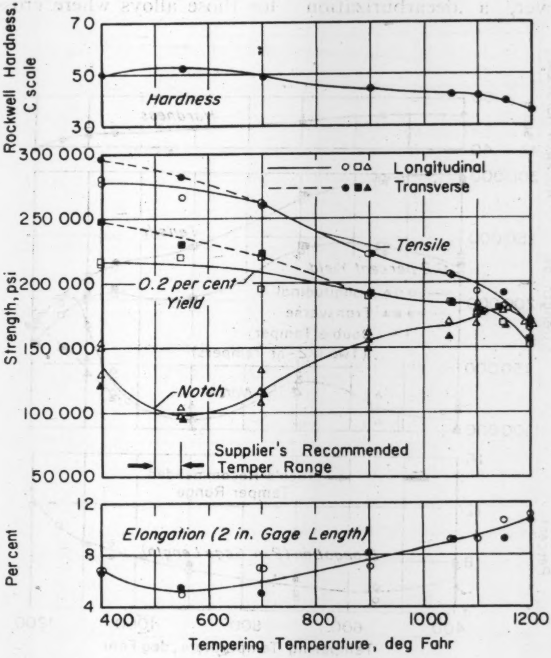


FIG. 6.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for UHS 280 Air Melt 0.063-in. Sheet as a Function of Tempering Temperature.

alloys. This behavior is in contrast to the remainder of the ferritic alloys which exhibit sheet strengths very little different from bar stock of essentially equal carbon content. It might be expected that these differences in yield strength between bar stock and sheet of different thicknesses are associated with decarburization. However, a decarburization study did not confirm this (see Appendix II).

The elongations, in general, are quite insensitive to those factors which affect the strength. All the ferritic steels with the exception of Vascojet 1000 exhibit elongations between 6 and 8 per cent at the lowest tempering temperature and between 12 and 15 per cent at the highest tempering temperatures investigated. The elongation of Vascojet 1000 at the lowest tempering temperature is less than 3 per cent.

Regarding directionality effects, it is noted that with the exception of Vascojet 1000 and UHS 280 there is practically no difference between the longitudinal and transverse strength properties and the elongations. This might be expected for those alloys where cross rolling was applied. An unexpected result is the higher transverse strengths and lower elongations observed for cross-rolled Vascojet 1000 and the higher transverse strengths observed for the cross-rolled UHS 280 at tempering temperatures below about 800 F

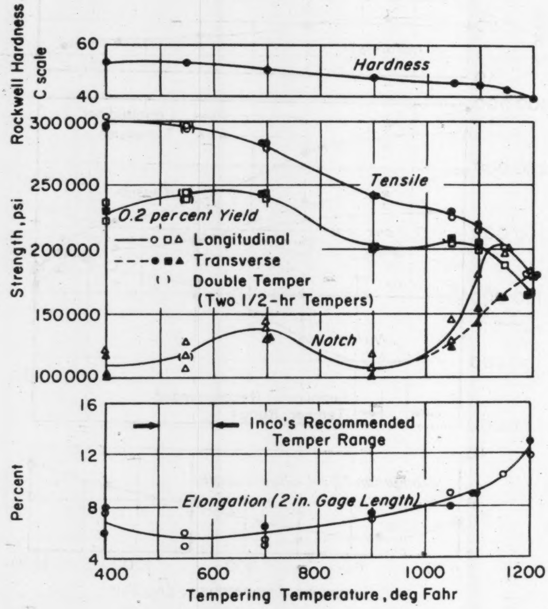


FIG. 7.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for 300 M Air Melt 0.063-in. Sheet as a Function of Tempering Temperature.

Sharp Notch Properties of Ferritic Steels:

The most important observation here is that notch sensitivity is observed for all alloys in the conventional temper range. Another important observation is the great sensitivity of the sharp edge-notch test to the well-known regions of embrittlement. Thus, the "blue brittle" (500 to 600 F) region for SAE 4340 steel (Fig. 2) is clearly delineated by a pronounced drop in the notch strength. The same is observed for the 850 F embrittlement of the martensitic stainless steels (Figs. 10 and 11).

The low-alloy ferritic steels SAE 4340 and AMS 6434 exhibit notch sensitivity only in a range of tempering temperatures up to about 700 F. This corresponds to the above-mentioned blue brittle range. The effect is largest for SAE 4340 and very small for consumable electrode vacuum-melted AMS 6434. A similar pronounced minima in notch strength at 550 F is noted for UHS 280. Both 300 M and X-200 alloys contain sufficient amounts of silicon to shift the minima in notch strength to higher tempering temperatures. This shift is most pronounced for 300 M alloy which has the higher silicon content. This is an effect which has been reported previously for the impact strength and tensile ductility of silicon-containing steel by Alten and Payson (20), Sands (21), and Shih, Averbach, and Cohen (22). For such steels a maximum in the impact strength is noticed for about 500 to 600 F temper-

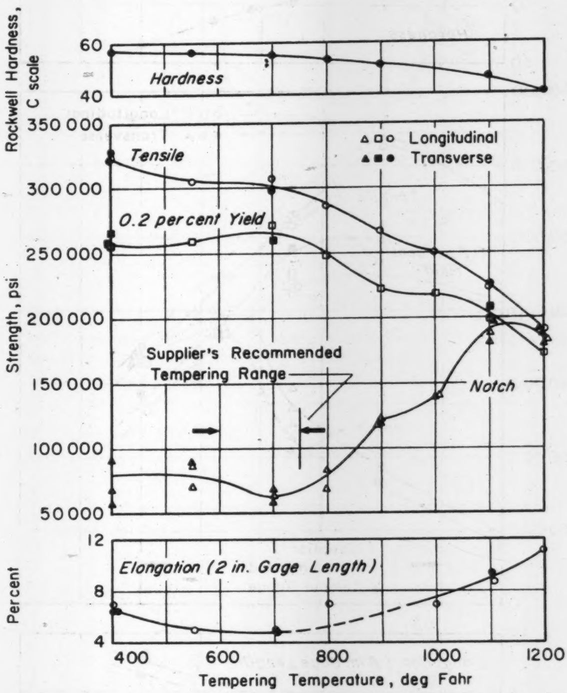


FIG. 8.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for X-200 Air Melt 0.063-in. Sheet as a Function of Tempering Temperature.

ing, and this is generally the recommended temperature range. It will, however, be noted (Figs. 7 and 8) that no substantial recovery of the sharp notch strength occurs in this region for 300 M and X-200 alloys. In contrast to the 4340 types, the three higher carbon high-bred steels, UHS 280, 300 M, and X-200, exhibit a broad region of notch sensitivity which extends to about 1100 F tempering temperature. In the case of 300 M alloy (Fig. 7), a distinct minima is observed at 900 F and for X-200 alloy (Fig. 8) an inflection occurs in the notch strength curve at about the same temperature. It will be noted that this temperature also produces a corresponding inflection in the tensile yield and ultimate strengths. This would suggest that the same carbide-forming elements giving

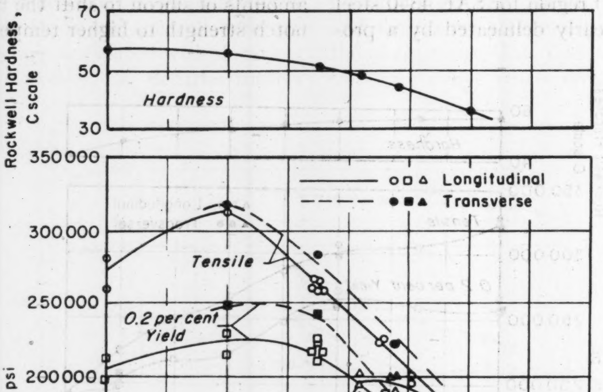


Fig. 9.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for Vascojet 1000 Air Melt 0.063-in. Sheet as a Function of Tempering Temperature.

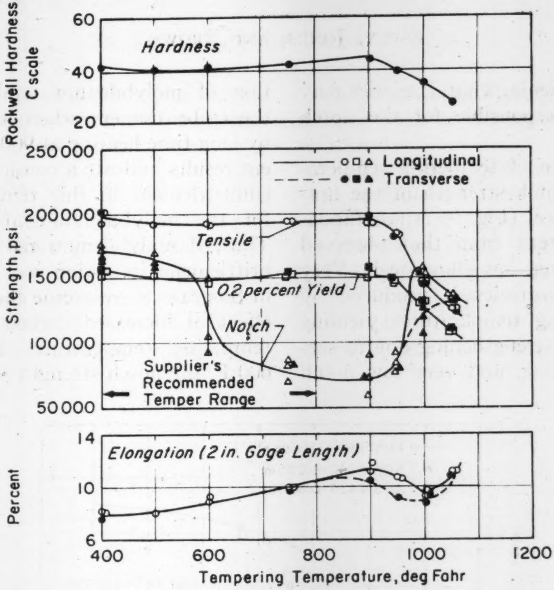


FIG. 10.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for AISI 410 Air Melt 0.063-in. Sheet as a Function of Tempering Temperature.

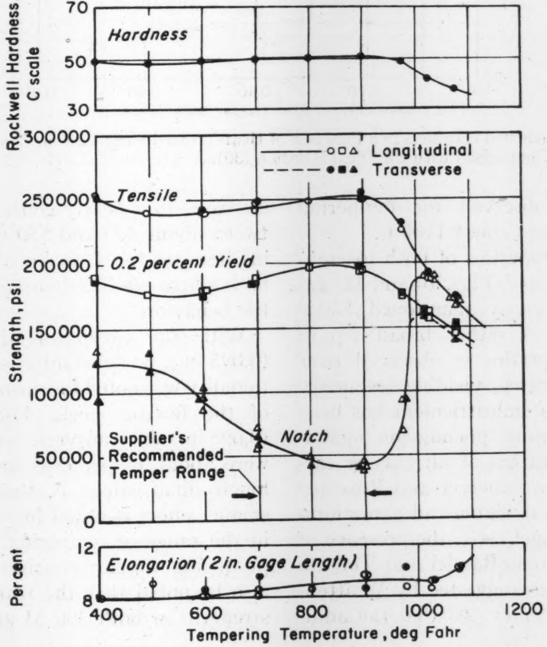


FIG. 11.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for 12 MoV Air Melt 0.063-in. Sheet as a Function of Tempering Temperature.

secondary hardening characteristics may be, in part, responsible for the notch sensitivity.

The influence of tempering temperature on the notch strength of the hot-worked tool steel (Fig. 9) is not fundamentally different from that observed for the high-bred low-alloy steels. Very high notch sensitivity is produced by those tempering temperatures yielding the maximum strengthening due to secondary hardening, and very low notch sensitivity is observed for tempering temperatures over about 1100 F.

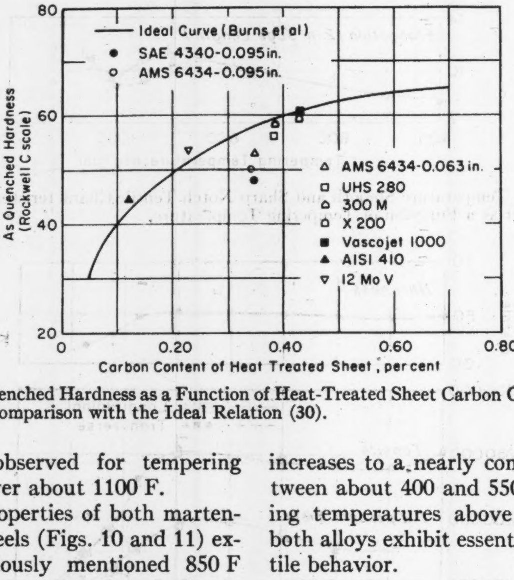


FIG. 12.—As-quenched Hardness as a Function of Heat-Treated Sheet Carbon Content for Various Ferritic Steels in Comparison with the Ideal Relation (30).

The notch properties of both martensitic stainless steels (Figs. 10 and 11) exhibit the previously mentioned 850 F embrittlement. A rather broad dip in the notch properties is observed over the tempering range yielding secondary hardening. This embrittlement has been ascribed to various phenomena including the precipitation of nitrides or carbonitrides (Whittenberger and Rosenow (23)); formation of sigma and chromium-rich ferrite (Hagel (24)); the presence of excess phosphorus (Bandel and Tofaute (25)). It has been suggested by Whittenberger and Rosenow (23) that the addition of molybdenum tends to reduce the embrittlement when it is produced by long-time heating at 900 F. The present results indicate a considerably larger embrittlement in this tempering range for the molybdenum-containing alloy. Thus, if molybdenum reduces the embrittlement, its action was not sufficient in this case to overcome the detrimental effect of increased carbon content. At tempering temperatures below about 600 F, the notch strength of these steels increases to a nearly constant value between about 400 and 550 F. At tempering temperatures above about 1050 F, both alloys exhibit essentially notch ductile behavior.

With the exception of AMS 6434 (0.095 in.), no substantial effect of directionality was noted for the notch strength of the ferritic steels. For AMS 6434 (0.095 in.) the transverse notch strengths were about 15 per cent lower than the longitudinal values. A similar but much smaller effect is noted for Vascojet 1000 in the range of tempering temperatures producing notch sensitivity. It should also be noted that the transverse notch strengths of both 300 M and X-200 are somewhat lower than the longitudinal values at the highest tempering temperatures.

Smooth Properties of Precipitation-Hardening Stainless Steels:

The tensile and yield strength and hardness of these materials (Figs. 13 to 15) pass through a maximum as the tempering temperature increases. This is due to the formation of an aging type precipitate. The exact nature of this precipitate appears to be, as yet, unknown, but it may be associated with the presence of aluminum or nitrogen. The lowest tempering temperature employed for AM 350 produced a fully aged structure. Generally, the suppliers recommended tempering temperatures for these alloys produce either a fully or overaged structure.

The longitudinal elongations of 17-7 PH and PH 15-7-Mo alloys exhibit shallow minima in the tempering region producing maximum strength. For AM 350 alloy, the elongation appears to exhibit a maximum at 900 F and then decreases abruptly with decreasing tempering temperature.

A definite but small directionality effect is exhibited by the smooth strength values of PH 15-7 Mo alloy and by the elongations of both 17-7 PH and PH 15-7 Mo alloys. These effects may be explained in part by ferrite stringers present to varying degrees in this class of alloys. Where directionality effects are observed, the strengths in the transverse direction are higher and the elongations lower.

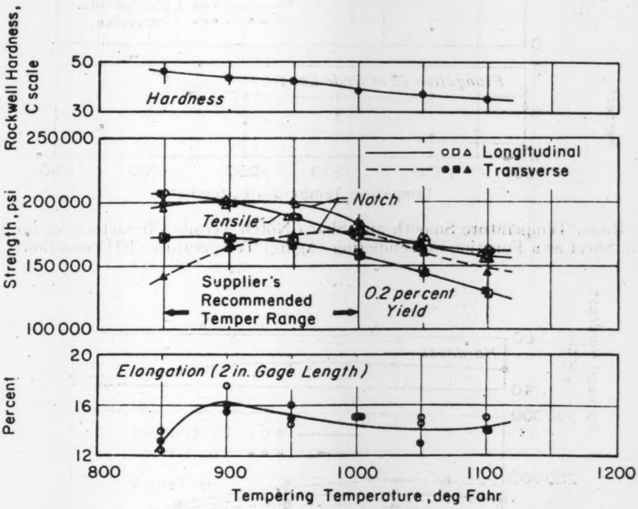


FIG. 13.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for AM 350 Air Melt 0.063-in. Sheet as a Function of Tempering (Aging) Temperature (SCT conditions).

Notch Properties of Precipitation-Hardening Stainless Steels:

Notch sensitivity in these alloys apparently develops in a manner not entirely related to aging. For AM 350 alloy (Fig. 13), the longitudinal notch strength is nearly equal to the tensile strength over the entire temper range. A slight maximum in notch strength is observed between 900 and 950 F. For PH-15-7 Mo

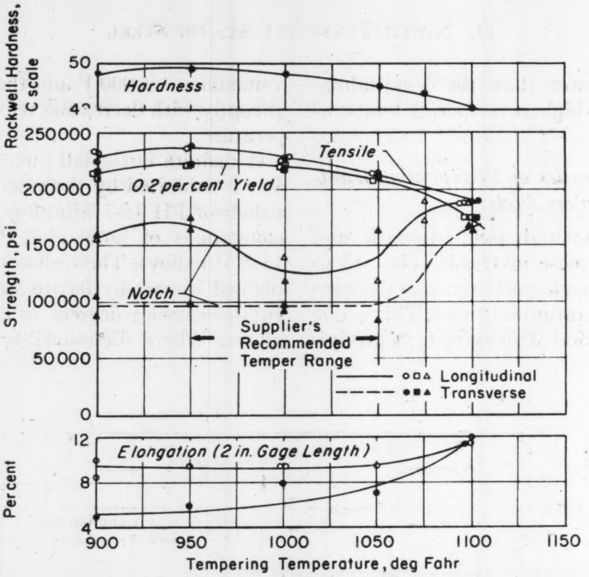


FIG. 14.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for 17-7PH Air Melt 0.063-in. Sheet as a Function of Tempering (Aging) Temperature (RH conditions).

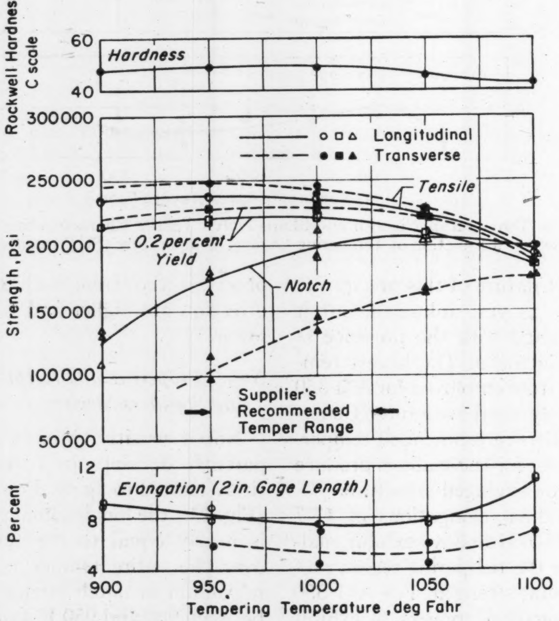


FIG. 15.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for PH 15-7 Mo Air Melt 0.063-in. Sheet as a Function of Tempering (Aging) Temperature (RH conditions).

alloy (Fig. 15), the notch strength in both directions decreases continuously with decreasing tempering temperature below about 1050 F. Complex and unexpected trends (Fig. 14) are observed for 17-7 PH alloy. The longitudinal notch strength exhibits a minimum between tempering temperatures of 1000 and 1050 F and passes through a maximum at a temperature of about 925 F. The authors have no explanation for this peculiar behavior and feel that additional tests are necessary to establish it as a true characteristic of the alloy.

The low notch strengths in PH 15-7 Mo and 17-7 PH alloys may, in part, be due to the presence of substantial quantities of free ferrite. This might be inferred from the known (26) deleterious effect of intermediate transformation products on the notch properties of tempered martensites.

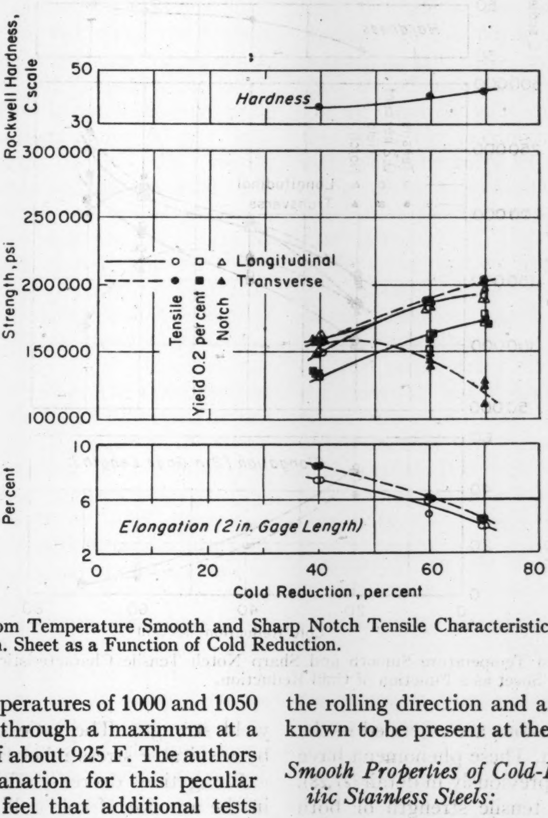


FIG. 16.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for AISI 304L Air Melt 0.063-in. Sheet as a Function of Cold Reduction.

The pronounced inferiority of the transverse notch strengths of all three of these alloys undoubtedly is, in part, associated with the presence of the ferrite stringers. These are highly elongated in the rolling direction and a precipitate is known to be present at their boundaries.

Smooth Properties of Cold-Rolled Austenitic Stainless Steels:

The primary variable controlling the strength of AISI type 304L and 301 steels is the amount of cold rolling. This cold reduction produces several effects other than strain hardening which can influence the mechanical properties, namely: (1) residual stresses of both the first and second order increase with the amount of cold rolling; (2) martensite is formed in the type 301 steel and possibly to a small extent in the type 304 steel; (3) the amount of transformation increases with the cold reduction; and (4) grains and inclusions are extended in the rolling direction. These phenomena have been discussed previously in detail (27,28).

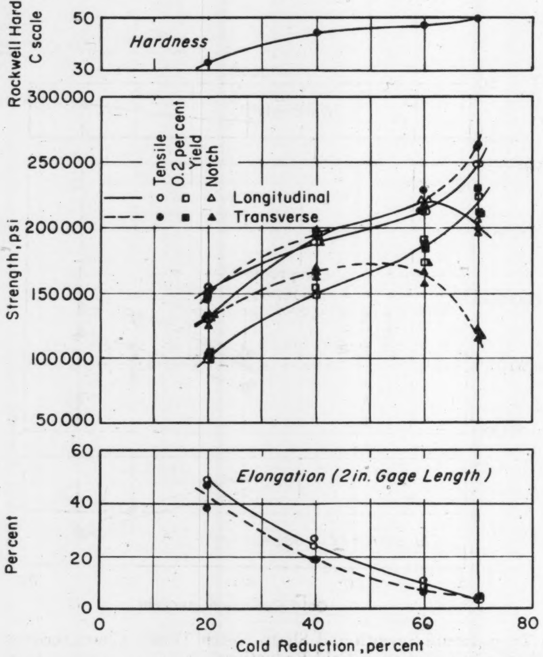


FIG. 17.—Room Temperature Smooth and Sharp Notch Tensile Characteristics for AISI 301 Air Melt 0.063-in. Sheet as a Function of Cold Reduction.

The smooth tensile strength of both alloys (Figs. 16 and 17) increases rapidly with the amount of cold reduction, the most pronounced strengthening effect being observed for AISI type 301 steel at higher orders of cold reduction. This large effect is primarily explained by martensite formation during rolling and subsequent testing. The yield strength exhibits generally similar trends.

The yield-tensile ratio for these alloys is quite low, as might be expected since the residual stresses tend to depress the yield strength. The ratio can be raised by a suitable stress relief treatment (28).

Elongations decrease with an increase in the amount of cold rolling and reach values of only 2 or 3 per cent for AISI type 301 steel, cold reduced 70 per cent.

The influence of directionality on the tensile strength is similar for both alloys, the transverse direction being slightly stronger for sheet cold rolled above about 40 per cent. In contrast, essentially no directionality is observed in the yield strength for either alloy.

Notch Properties of Cold-Rolled Austenitic Stainless Steels:

The notch sensitivity in these alloys depends strongly on the testing direction. In the longitudinal direction, essentially notch ductile behavior for AISI type 304L (Fig. 16) is observed up to the highest cold reduction investigated (70 per cent). For AISI type 301 (Fig. 17), the longitudinal direction is notch-sensitive only for cold reductions above 60 per cent. Notch strengths below the tensile strength are observed also for AISI type 301 at 20 per cent cold reduction. However, these low values are not associated with brittle behavior. In this relatively soft condition, cracking at the notch root occurs at low loads, probably due to strong localized martensite formation produced by the high plastic strain gradient at the notch. The crack runs in a ductile manner with large plastic deformations preceding it across the specimen. With increasing cold reduction, the tendency to early cracking fades out presumably because of the increasing amount of martensite formed during cold rolling and the consequent reduction of the martensite gradient at the notch root.

In the transverse direction, pronounced notch sensitivity is observed for both alloys. In the case of AISI type 304L it develops rapidly for cold reductions greater than 40 per cent. For AISI type 301, the trend of the transverse notch strength curve is similar to that for the longitudinal direction. However, particularly at the higher values of cold reduction the transverse notch strength is very much lower than that of the longitudinal direction. No definite explanation is offered for this high transverse notch sensitivity. It undoubtedly is associated with the severely cold-worked structure and possibly also with inclusions.

COMPARISON OF ALLOYS

The previously discussed results make possible a comparison of the notch sensitivity of the alloys at various strength levels. Additionally, a similar comparison may be made on the basis of the yield strength. In this paper the ratio of the notch strength to the tensile strength is used as indication of notch sensitivity. For an infinitely wide sheet, notched at the edges, the maximum value of this ratio would be unity for material undergoing sufficient plastic flow to eliminate the initial elastic stress concentration. The geometry of the specimen used in this investigation results in a slight strengthening for such completely notch ductile behavior. The actual maximum sharp notch strength ratio will depend on the stress-strain characteristics of the material, but it appears to be between 1.00 and 1.05 for the alloys tested in this investigation. For practical purposes it is considered that sharp notch ratios less than 1.00 indicate notch sensitivity.

Comparison on the Basis of Strength Level:

Sharp notch strength ratio as a function of the tensile strength is shown in Figs. 18 to 21 for the various alloys. The curves generally represent the longitudinal notch properties, and only where substantial directionality was observed is the transverse sharp notch strength ratio shown.

The trend of the sharp notch strength ratios with increasing strength level for these sheet alloys is not different fundamentally from that previously derived from tests on circumferentially-notched cylindrical specimens (**12,14**). Thus, three distinct ranges of behavior are observed as follows: (1) at low strength levels the sharp notch strength ratio remains essentially constant or decreases slowly;[7] (2) in a narrow range of higher strength levels the sharp notch strength ratio decreases rapidly to low values; and (3) at the highest strength levels the sharp notch strength ratio continues to fall but at a decreasing rate, or may exhibit minima corresponding to certain embrittlements associated with the heat treatment. Depending on the alloy, one or more of these ranges of behavior are observed.

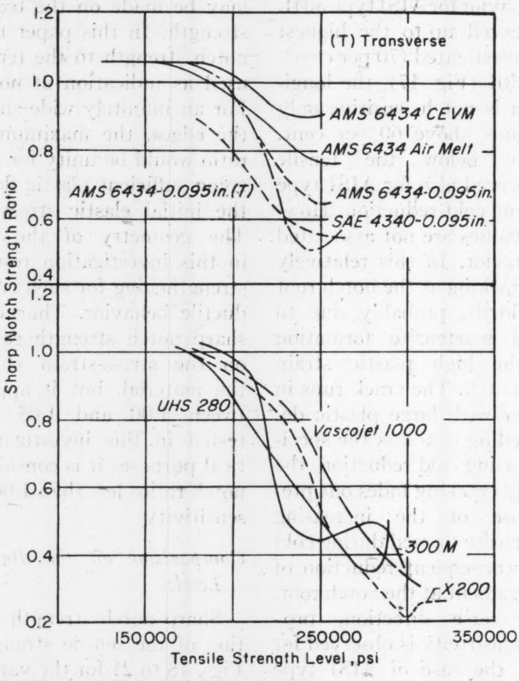


FIG. 18.—Relation Between Sharp Notch Strength Ratio and Tensile Strength Level for Various Ferritic Steels Tested at Room Temperature.

Considering first the low-alloy ferritic steels (Fig. 18), it is noted that completely notch ductile behavior is obtained only at tensile strength levels below about 180,000 psi. The trend of the sharp notch strength ratio curve with increasing strength level depends strongly on the thickness. Thus, in AMS 6434 the 0.095-in. sheet develops notch sensitivity much more rapidly than 0.063-in. sheet, and at high strength levels the 0.095-in. sheet exhibits considerably lower sharp notch strength ratios. It is noted that consumable electrode vacuum melt AMS 6434 is noticeably superior to the air melt. The vacuum-melted material exhibits a sharp notch strength ratio of 0.9 at 250,000 psi. An interesting observation is the higher notch sensitivity in the transverse direction observed for the cross-rolled AMS 6434 steel. The development of notch sensitivity in the high-bred alloys and the tool steel is quite different. Thus, 300 M shows a very rapidly decreasing sharp notch strength ratio in a narrow range of strength levels between 210,000 and 230,000 psi while notch sensitivity in Vascojet 1000 develops less rapidly. Strength levels over 300,000 psi can be obtained with these alloys. However, it should be noted that these high strength conditions are extremely brittle.

---

[7] For tests on circumferentially notched cylindrical specimens, the corresponding behavior results in a notch strength ratio of approximately 1.5 due to the transverse constraints.

The results for the martensitic stainless steel, AISI type 410 (Fig. 19), emphasize that severe embrittlement occurs even at relatively low strength levels (<180,000 psi) and that the least notch-sensitive high-strength conditions are obtained by tempering at less than 550 F. The modified martensitic stainless 12 MoV steel (Fig. 19) represents a very substantial improvement over AISI type 410. However, severe notch sensitivity is still associated with the highest strength levels. Again, the least notch-sensitive high-strength condition is produced by tempering at less than 550 F.

The precipitation-hardening stainless

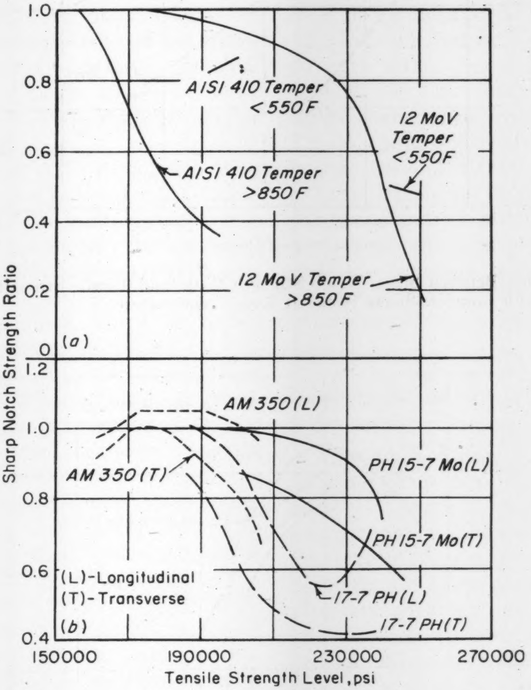


FIG. 19.—Relation Between Sharp Notch Strength Ratio and Tensile Strength Level for Stainless Steels at Room Temperature.

(a) For two martensitic stainless steels.

(b) For several precipitation hardening austenitic stainless steels.

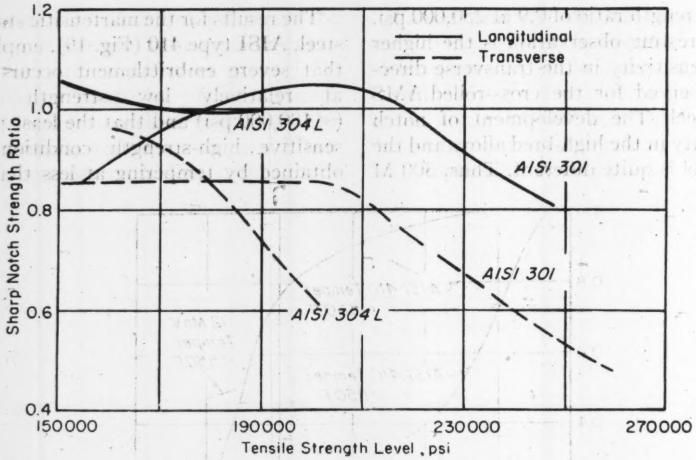


FIG. 20.—Relation Between Sharp Notch Strength Ratio and Tensile Strength Level for Two Cold-Rolled Austenitic Stainless Steels Tested at Room Temperature.

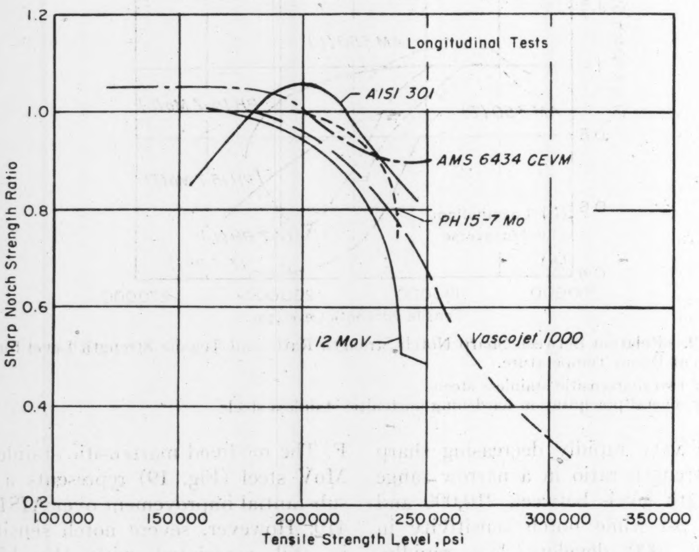


FIG. 21.—Comparison of Various Sheet Alloys on the Basis of the Room Temperature Sharp Notch Strength Ratio.

steels are compared in Fig. 19.[8] At strength levels of less than 190,000 psi, very low notch sensitivity characterizes all these alloys in the longitudinal direction. Strength levels over 210,000 psi are achieved only for the 17-7 PH and PH 15-7 Mo materials. In this range, PH 15-7 Mo appears to be superior to 17-7 PH. A most important observation is the increased notch sensitivity of the transverse as compared with the longitudinal direction. Based on the transverse notch properties PH 15-7 Mo is again superior to the 17-7 PH.

The results for AISI type 304 L (Fig. 20) indicate that essentially notch ductile behavior in the longitudinal direction is maintained to the highest strength level produced by cold reduction (190,000 psi). For AISI type 301 tested in the longitudinal direction, complete notch ductile behavior is observed up to 210,000 psi.[9] At higher strength levels the longitudinal sharp notch strength ratio decreases rather slowly and is about 0.8 at 245,000 psi. As was observed for the precipitation hardening austenitic stainless steels, the transverse direction is much more notch-sensitive than the longitudinal. This difference apparently is still more pronounced for AISI type 304 L.

It is of interest to compare representative alloys from each group, as shown in Fig. 21. It is difficult to make generalizations regarding the relative performance of the various alloys at those strength levels where the sharp notch strength ratios differ only slightly. In such cases, heat to heat differences may reverse the order of performance. However, several observations of a general nature appear to be fairly well established, namely: (1) With the exception of AISI type 301, completely notch ductile behavior is observed only at strength levels somewhat less than 200,000 psi. For AISI type 301 (longitudinal) this behavior is maintained up to about 210,000 psi. (2) At strength levels between 200,000 psi and 250,000 psi the most brittle behavior is observed for the 12 MoV steel while high sharp notch strength ratios characterize AMS 6434 CEVM. (3) Low sharp notch strength ratios characterize strength levels over 275,000 psi as represented by the behavior of Vascojet 1000.

Attention should be directed again to the low transverse notch strength ratios of the austenitic precipitation hardening and the straight austenitic stainless steels. These are below the longitudinal values for all the alloys represented in Fig. 21.

Comparison on the Basis of Yield Strengths:

As mentioned previously, the authors do not believe that the yield strength alone is always a good indication of strength potential. However, because of the present emphasis placed on it by designers, the alloys have been compared at several yield strength levels in Table IV. These levels are representative of present and future goals in the design of thin-walled pressure vessels. In the formulation of this table, the alloys have been rated in accordance with their sharp notch strength ratios in the longitudinal testing direction. The transverse sharp notch strength ratios are shown only when they differ substantially from the longitudinal values.

At the 180,000 psi yield level, all of the alloys exhibit low notch sensitivity in both the longitudinal and transverse direction, with the exception of the 12 MoV alloy. For this alloy, the notch strength ratio varies from 0.25 to 0.50,

---

[8] In this representation only the "fully aged" or "over aged" conditions are considered. Underaging appears to increase the notch sensitivity (see Figs. 14 and 15).

[9] As discussed previously the decrease in notch strength ratio for AISI 301 steel at strength levels below 190,000 psi is not associated with brittle behavior.

TABLE IV.—RATINGS OF THE ALLOYS AT VARIOUS YIELD STRENGTH LEVELS ACCORDING TO THE SHARP NOTCH STRENGTH RATIO.

0.2 Per Cent Yield Strength, psi	Alloy[a]	Sharp Notch Strength Ratio	Sheet Direction, Longitudinal or Transverse	Temper Condition
180×10^3	AISI 301	1.04	L	58 per cent cold rolled
		0.79	T	
	AMS 6434 CEVM	1.02	L and T	850 F
	AMS 6434, 0.095-in.	1.00	L	950 F
		0.84	T	
	300 M	1.00	L	1160 F
	17-7 PH	1.00	L	1095 F
		0.84	T	
	4340	0.95	L and T	825 F
	AMS 6434	0.95	L and T	800 F
	X-200	0.95	L and T	1175 F
	Vascojet 1000	0.90	L	1130 F
	UHS 280	0.89	L and T	1100 F
	12 MoV	0.50	L and T	450 F
		0.37	L and T	625 F
		0.25	L and T	960 F
210×10^3	AMS 6434 CEVM	0.90	L and T	400 F
	PH 15-7 Mo	0.90	L	1050 F
		0.74	T	
	AISI 301	0.90	L	68 per cent cold rolled
		0.55	T	
	X-200	0.81	L and T	1075 F
	AMS 6434	0.77	L and T	400 F
	AMS 6434, 0.095-in.	0.70	L	600 F
		0.59	T	
	17-7 PH	0.65	L	1050 F
		0.46	T	
	Vascojet 1000	0.58	L	1080 F
	SAE 4340	0.51	L and T	500 F
	UHS 280	0.41	L and T	650 F
	300 M	0.41	L and T	850 F
225×10^3	AISI 301	0.75	L	72 per cent cold rolled
		0.40	T	
	PH 15-7 Mo	0.68	L	1035 F
		0.35	T	925 F
	300 M	0.47	L and T	775 F
	X-200	0.43	L and T	890 F
	UHS 280	0.39	L and T	650 F
	Vascojet 1000	0.35	L	1025 F
240×10^3	300 M	0.49	L and T	700 F
	300 M	0.40	L and T	500 F
	UHS 280	0.36	T	500 F
	X-200	0.31	L and T	830 F

[a] 0.063 in. thick sheet unless otherwise stated.

depending on the tempering temperature selected. At 210,000 psi yield strength level, low notch sensitivity characterizes only a few of the alloys and pronounced differences between the longitudinal and transverse properties are observed. At the 225,000 and 240,000 psi yield levels, all the alloys exhibit high notch sensitivity, with the exception of AISI type 301 (longitudinal).

COMPARISON OF DIFFERENT TYPES OF TESTS

As previously mentioned, a number of test procedures have been proposed for evaluating the tendency of any given metal condition to fail in a brittle manner. It is of the utmost importance that the results from these various tests be compared in order to select the most suitable test procedure and to gain an understanding of those factors which control the materials response to high stress concentrations.

TABLE V.—COMPARISON OF SHARP EDGE NOTCH RESULTS WITH THOSE OBTAINED USING SRAWLEY'S CENTER CRACK SPECIMEN.

Alloy	Tensile Strength, psi	Sheet Direction	Edge Notch Thickness, in.	Center Crack Thickness, in.	Sharp Edge Notch Strength Ratio	Center Crack Strength Ratio[a]	Srawley's FATT[b], deg Fahr
Vascojet 1000	270×10^3	L	0.063	0.063	0.46	0.46	200
Vascojet 1000	250	L	0.063	0.090	0.71	0.71	225
300 M	275	L	0.063	0.083	0.50	0.69	100
12 MoV	[c]250	L	0.063	0.041	0.16	0.44 (0.15–0.20)	300
AISI 410	[d]190	L	0.063	0.039	0.35	1.05 (0.65–0.70)	...
AISI 410	[e]190	L	0.063	0.039	0.65 (1.03)	1.05	0
17-7PH	225	L	0.020	0.10	[f]0.75	1.00	50
17-7PH	225	L	0.020	0.020	[f]0.75	[g]0.82	...
		T	0.020	0.020	[f]0.49	[g]0.65	...
17-7PH	196	L	0.020	0.020	[f]0.87	[g]0.87	...
		T	0.020	0.020	[f]0.82	[g]0.86	...
PH 15-7 Mo	235	L	0.063	0.042	0.85	0.71	...
		T	0.063	0.042	0.60	0.73	...
PH 15-7 Mo	207	T	0.063	0.042	0.85	0.87	...

[a] Data obtained by Srawley unless otherwise indicated.
[b] Fracture appearance transition temperature.
[c] 900 F temper.
[d] 825 F temper.
[e] 600 F temper.
[f] Unpublished data by authors.
[g] Data from Armco Steel Corp. Research Laboratories, Middletown, Ohio.
( ) Values corrected for thickness effect.

Evaluation of ultra-high-strength sheet alloys in various programs has been started only recently and therefore only a limited amount of information is available. However, the various investigators are in close contact and where possible are working with the same heats. At the present time a sufficient amount of data is available from Srawley's investigations (8,29) to permit comparisons. Srawley employs a center crack specimen, ½-in. wide with a ratio of crack length to specimen width varying from about 0.30 to 0.50. Cracks were introduced by loading fully heat-treated hydrogen-embrittled specimens at very low stresses. The hydrogen was then removed by baking. Some data are also available for specimens cracked in fatigue.

Results at room temperature obtained for several alloys, using both the sharp edge notch and the center crack specimen, are shown in Table V. Notch strength ratios for the sharp edge notch test and corresponding center-crack strength ratios for Srawley's test are given. Also included are values of the fracture appearance transition temperature (FATT) reported by Srawley (29). Comparisons are made on the basis of equal strength level rather than for identical heat treatments. This is necessary since the same heats were not tested in both investigations. Thus, small differences in composition resulted in somewhat different smooth strengths for the same heat treatment. Unfortunately, a comparison for the same sheet thicknesses is not always possible. However, it would generally be expected that thicker sheet would exhibit greater embrittlement.

The geometry of the two types of specimens is such that approximately the same initial elastic stress concentration is produced. If this were the major variable controlling the strength, then for brittle fracture the center crack strength ratio and the sharp notch strength ratio should be equal at the same strength level. According to Table V such an ideal behavior is observed for Vascojet 1000 (0.063-in.) and for the majority of tests on 17-7 PH (0.020-in.). However, discrepancies are observed for the remaining alloys with the sharp edge notch test indicating greater embrittlement. For some cases this may be explained by the thickness effect. This effect has been established by Srawley (8) for the 12 MoV alloy (900 F temper) over a thickness range between 0.010 to 0.050 in. Using a correction factor derived from these data,[10] the 0.063-in. thickness center crack strength ratios for both 12 MoV and AISI type 410 are also shown in Table V. The corrected values are in satisfactory agreement with the sharp notch strength ratios. However, the thickness effect could not explain the greater embrittlement indicated by the sharp edge notch test for 300 M and for 17-7 PH (tested by Srawley), since it is in the wrong direction. Furthermore, it is felt that the difference in results for AISI type 410 (825 F temper) is too large to be explained by a thickness effect alone. This alloy condition shows essentially complete notch ductile behavior in the center crack test. This is most surprising since this tempering temperature is known to produce high embrittlement. The large magnitude of this embrittlement is quite evident from the results of the sharp edge notch test (see Fig. 10).

It is of interest to compare the rating of the various alloys on the basis of sharp notch strength ratio, center crack strength ratio, and FATT. It is noted that both the sharp notch strength ratio and the center crack strength ratio rate the alloys in the same order. Using the FATT values, the rating is somewhat different. This is not unexpected since the FATT's have not been accurately established in some cases. However, there is no question that all three methods agree in their definition of the best and worst material conditions.

Srawley (29) implies that natural cracks produce higher embrittlement than machined notches. Tests were made on 0.039-in. thick AISI type 410 tempered at 600 F. The center cracks were produced by hydrogen embrittlement and by fatigue. In addition, a center crack was simulated by a 0.002-in. radius Elox machined notch. The large difference (Table VI) between the sharp edge notch strength and the strengths of the other specimens, at room temperature, is almost entirely accounted for by the previously discussed thickness effect. However, for the low-temperature tests, the strength of the sharp edge notch specimen is lower by a factor of 10 than the strength of the Elox center-notched specimen. In fact, the latter specimen indicates a transition temperature below −300 F. These discrepancies are too large to be explained by any known thickness effect. Thus, it appears that Srawley's data, using Elox center notches, does not prove that natural cracks have greater effectiveness than very sharp machined edge notches. In fact, all of the data in Table V indicate the embrittlement produced by the sharp edge notch equals or exceeds that produced by the hydrogen induced center crack.

---

[10] These data define a nearly linear decrease in net fracture stress with increasing thickness between 0.010 in. and 0.050 in. The corresponding rate is approximately 3000 psi per mil.

The claim has also been made (8) that in edge notch specimens bending moments cannot be avoided due to the unsymmetrical nature of fracture development. On the other hand, fracture in the center crack specimen is presumed to proceed in a more symmetrical manner. The authors agree that the center crack specimen inherently tends to cause symmetrical crack development. However, there is no evidence that extraneous bending moments influence the results of concentrically loaded edge notch specimens. If eccentricity were a significant factor it should result in excessive scatter since it would vary from test to test in an uncontrolled manner. However, no more scatter is noted for the edge notch test than for the center-cracked test.

PRACTICAL SIGNIFICANCE OF THE RESULTS

Rather than present a list of formal conclusions, it appears more desirable to attempt to set forth the practical significance of the results, as an aid to fabricators, designers, and alloy producers. In doing this the authors have drawn, in part, on experience gained from application of ultra-high-strength steels in the aircraft industry. Additionally, where possible, use is made of information developed from the investigation of bar stock sharp notch properties.

It should be realized that conclusions from the present results apply directly only to room temperature, to the loading rates, compositions and the thicknesses studied. Furthermore, it is important to point out that the weldability of the various alloys has not been investigated and such information is needed for a complete evaluation of these materials.

Significance in Design and Fabrication:

Considerable confusion currently exists as to the actual meaning of strength values derived from sharp notch tests or tests employing natural cracks. It is not possible at this time to use the actual notch strength values in design, except perhaps to indicate the "worst" conditions. The application of fracture dynamics theory (5,6) does yield critical crack lengths for a given alloy condition and average stress field in the component. However, the applicability of this theory to metal fracture is, as yet, not definitely established. Furthermore, for very high-strength levels critical cracks are so small as to be very difficult to detect. Unfortunately, areas of essentially zero ductility or networks of fine cracks may not be revealed by any known nondestructive inspection technique.

The fabricator and designer should, however, use the sharp notch strength

TABLE VI.—COMPARISON OF RESULTS OBTAINED USING SEVERAL TYPES OF SPECIMENS FOR AISI TYPE 410 TEMPERED AT 600 F.

Type of Test	80 F Strength, psi	−320 F Strength, psi
Sharp edge notch	125×10^3	20
H_2 center crack	200	...
Fatigue center crack	200	...
Elox center notch	200	210[a]

[a] Extrapolated from −290 F.

ratios as a guide for selecting materials and defining the seriousness of the problem. If an alloy shows completely notch ductile behavior (notch strength ratio greater than unity for the present test), there is only a small danger of brittle failure in a reasonably designed component subjected to static stresses approaching the yield strength. With the exception of AISI type 301, none of the alloys investigated exhibits completely notch ductile behavior at strength levels appreciably above 180,000 psi. This limit is extended to about 210,000 psi for cold-rolled AISI type 301 but only in the longitudinal direction.

Particular attention should be given to the shape of the sharp notch strength ratio *versus* strength level (or yield level) curve. Strength levels near the rapidly descending portion of the curve should be used with caution even if possessing high notch strength ratios. In such cases, a small unintentional deviation in the tempering temperature may greatly increase the embrittlement. At strength levels up to 250,000 psi there appears to be no advantage in using the higher alloyed materials at the 0.40 carbon level, essentially equal or superior results being obtained with consumable electrode vacuum melt AMS 6434. At strength levels in excess of 275,000 psi all the investigated alloys exhibit very high notch sensitivity. This does not necessarily mean these conditions are useless. However, application of such notch-sensitive materials requires extremely careful attention to design and fabrication technique in order to avoid cracks resulting from heat treatment and welding, as well as from section discontinuities. For example, large welded airplane landing gears are now successfully fabricated from forgings at a strength level in excess of 260,000 psi. However, this was accomplished only after an intensive research and development program lasting several years and making use of the sharp notch tension test. Until better alloys are available, attempts should be made to raise the strength level in relatively small steps employing the best materials available and taking advantage of the beneficial effects of vacuum melting. This can be most rapidly and efficiently accomplished by making use of laboratory tests incorporating very high stress concentrations. These tests should be employed for evaluation of parent materials and welding techniques.

The present results emphasize that tests in the longitudinal direction alone may not be sufficient. High notch sensitivity appears to characterize the transverse properties of the austenitic steels. In the application of such steels, the transverse sheet direction should be oriented in the direction of minimum stress. Where balanced biaxial tension is approached, the design must be based on the transverse properties.

Considerable attention has been given to the elongation. From the present results, there is no question that the elongation is quite insensitive to those factors affecting the notch sensitivity. Therefore, the elongation may increase in a region of tempering temperature where the notch strength passes through a pronounced minimum (for example, AISI type 410, Fig. 10; 12 MoV alloy, Fig. 11). If the elongation is only a few per cent there is, of course, reason to suspect that the alloy may be notch-sensitive. Thus, the most highly embrittled conditions of the ferritic alloys generally exhibit the lowest elongations. In contrast, AISI type 301 (longitudinal) at 70 per cent cold reduction (Fig. 17) has an elongation of only about 3 per cent but is not highly notch-sensitive. Furthermore, relatively high elongations do not necessarily correspond to notch ductile behavior (for example, AISI type 410, 900 F temper, Fig. 10).

It would appear that the yield strength or the yield-tensile ratio bears no direct relation to notch-sensitive behavior. A high yield strength is desirable only if coupled with satisfactory notch properties; otherwise brittle failure may occur at stresses well below the yield strength. A low yield strength - tensile strength ratio does not necessarily indicate the capacity to deform in the presence of high stress concentrations. Thus, all the high-bred steels (Figs. 6 to 8) and the martensitic stainless steels (Figs. 10 and 11) have relatively low yield-tensile ratios in the temper range producing very high notch sensitivity. In contrast, the SAE 4340 type alloys (Figs. 2 to 5) exhibit highly notch ductile behavior in the temper range where the yield-tensile ratio is very high. However, equally high yield-tensile ratios are observed for PH 15-7 Mo alloy (Fig. 15) at all tempering temperatures including those producing high notch sensitivity. Regardless of the yield-tensile ratio, if the alloy is notch ductile yielding at points of stress concentration can occur and will reduce the stress concentration.

It should be emphasized that when determining either the smooth or notch tensile characteristics, tests should be conducted on the actual sheet thickness employed. Furthermore, heat treatment of specimens should duplicate as closely as possible that to be used in the actual component.

Significance in Alloy Development:

Alloy steel development has been generally conducted with little attention to the effects of very high stress concentrations. The unfortunate result is that the newer alloys in sheet form are considerably more notch-sensitive than lower-carbon (0.30 to 0.35 carbon) grades of the old SAE 4340 type alloys.[11] Furthermore, without tests employing severe stress concentrations it is not possible to arrive at optimum heat treatments for a given composition. The recommended temper ranges for the various alloys have been added to Figs. 6 to 15. It is quite evident that frequently the recommended range coincides with those tempering temperatures producing maximum notch sensitivity.

For the purpose of alloy development there appears to be ample evidence that the sharp edge notch test is quite suitable for evaluating the sensitivity of a given sheet material condition to brittle fracture. The results are very reproducible even for highly notch-sensitive specimens. Presently, there does not appear to be any reason for assuming a natural crack will produce more significant data. The sharp notch test appears extremely sensitive to known embrittlements produced by the heat treatment. It is superior to the conventional impact test in this respect both because it is more sensitive to embrittling factors and because the derived strength values are directly comparable to the smooth tensile and yield strengths.

It is not possible to arrive at definite conclusions regarding chemical composition from the present data alone. A considerable amount of sharp notch data on bar stock (16) indicates required strength level should be obtained with the lowest possible carbon content. The same is probably true for sheet.

The introduction of high-silicon steels has resulted in considerably improved notch properties in the thickness direction of large forgings (17). Increases in the molybdenum content up to about 0.35 per cent and the addition of vanadium up to 0.20 per cent appear to improve the sharp notch strength of the SAE 4340 type alloys. The data are insufficient to indicate whether greater improvements would be produced by further increases in these elements. Similar effects of these alloying elements on 12 per cent chromium martensitic stainless steels are observed, however, the 850 F embrittlement is not appreciably reduced.

---

[11] Lower carbon grades of high-bred steels are now under development. A reduction in the carbon content would be expected to reduce the notch sensitivity. However, further tests will be necessary to determine whether these new steels are superior to AMS 6434 steel.

The exact role of inclusions and melting practice is as yet obscure. However, on the basis of the present results it may be stated that a low inclusion count is definitely desirable. In the SAE 4340 type alloys consumable electrode vacuum melting appears to be beneficial in increasing the notch strength in the 500 F embrittlement range. The reason for this is not clear; however, it is shown the vacuum melted alloy has a lower inclusion content.

High directionality in the notch properties and low transverse notch strength appears to be associated with those alloys having strongly directional microstructures. In the case of precipitation-hardening stainless steels, this possibly may be reduced by application of sufficient cross-rolling. It, however, is difficult to see how all directionality could be eliminated in the cold-rolled austenitic stainless steels.

Acknowledgments:

The authors are indebted to the U. S. Steel Corp., American Steel and Wire Division, for providing several of the alloys tested. Appreciation is also due W. Ruprecht, General Laboratories, American Car and Foundry Division, ACF Industries, Berwick, Pa. for his assistance in the carbon analysis and many helpful discussions regarding the general problem of thin-walled pressure vessel fabrication.

Thanks are due to G. Sachs of Syracuse University for his assistance in formulation of the program and many helpful discussions regarding interpretation of the data.

The authors are further indebted to A. J. Repko, J. L. Shannon, Jr., and G. Succop of the Nat. Aeronautics and Space Administration, Lewis Research Center, for their assistance in the testing program.

APPENDIX I

PROCEDURE FOR MACHINING THE SMOOTH AND SHARP-EDGE NOTCH SPECIMENS

The machining procedures outlined are designed to insure maintenance of the close tolerances specified in Fig. 1 and to produce a notch root radius not resolvable at 100× magnification.

While a jig bore would be desirable, all operations except notching can be performed on a good vertical milling machine. Specimens used at the National Aeronautical and Space Administration, Lewis Research Center are machined to specification in this manner. The specimen blank size is 2 by 8 in. and the blanks are usually in the solution-treated or annealed condition. However, using the techniques outlined, specimens have been produced from stock having a Rockwell hardness of 50 C. The operations may be conveniently divided into rough, finish, and notch machining. Except for notching, these procedures are identical for both specimen types. The exact rates of feed and speeds during machining will of course depend on the alloy and no definite rules can be set down. The lubricant is a commercial water-soluble oil recommended for machining stainless steel. Inspection of the finished notch specimen requires an optical comparator, preferably of 100× magnification.

Rough Machining:

Specimen blanks are generally machined in groups of five. The first step is to drill the loading pin holes $\frac{1}{16}$ in. under size. Finish boring accurately sizes these holes and positions them so that a maximum clearance of ±0.0002 in. exists between the holes and pins in a special jig. All subsequent machining is then done using this jig with the specimens clamped tightly together. The test section is milled with a four-flute end mill ($\frac{1}{4}$ to $\frac{3}{8}$-in. diam) to within $\frac{1}{8}$ in. of final size. The radius joining the test section is then cut with a boring bar to 0.005 in. over size. The test section is then carefully milled to blend with the radius. At this point, the test section is 0.010 in. over size.

Finish Machining:

All operations are now checked with indicators. The specimens remain in the pin jig. The head radius on one side is cut to size with a boring tool and the test section on the same side finished with an end mill. The specimens are then turned over on the pins and these operations repeated on the other side.

For smooth specimens, the head radius is carefully hand blended to the test section. This hand work is such that a slight taper (0.001 in.) is produced from each head to the center of the gage length. At this point the smooth specimens are complete.

Notching:

There are two procedures for roughing the notch depending on the hardness and work hardening characteristics of the alloy. For materials of less than about Rockwell hardness 30 C the notch is commonly tool cut. Specimens finished except for the notch are clamped in the pin jig and the notch roughed to about 0.135-in. depth using a standard 60-deg, 3-in. diameter milling cutter. The root radius is held to 0.010 in. or less at this point. Finishing is accomplished by fixing a Carbaloy or Kennametal tool in the head of the milling machine and drawing the specimen under the tool by movement of the bed. Generally, only one specimen at a time can be finished. The tool is diamond ground to a point and to within ±1 deg of the actual notch angle produced by the previous milling. The notch is then cut to approximately 0.005 in. of final depth. Up to this point no attempt is made to keep the radius sharp, a value between 0.004 and 0.005 in. being permissible. Generally, the notch may then be finished to size with one more cut, using a freshly ground tool. During the above described finishing procedure, the radius must be frequently examined with an optical comparator. This inspection will indicate when tool regrinding is necessary. It should be noted that when replacing the tool, particular attention must be given to proper tool alignment with respect to the notch. This can best be accomplished with a low-power microscope mounted on the milling machine.

Specimens having Rockwell hardnesses between 35 and 50 C, particularly in the austenitic grades, are rough notched by grinding. Use is again made of the pin jig which is fixed to the table of a surface grinder. It has been found that a root radius of from 0.005 to 0.007 in. can be produced with a 120 grit wheel dressed sharp to the 60-deg angle. Coolant is supplied both in the normal flood and through the wheel. Finishing then proceeds using the method outlined above, but is less time consuming because the ground notch is essentially at finished size.

The procedures described above were developed by Orin Manning of The Walters Co., Cleveland, Ohio. Mr. Manning points out that attempts to finish the notch by milling with Carboloy tipped cutters is extremely difficult. This difficulty is caused by the superimposed effect of small runouts in the cutter and in the arbor of the milling machine. These runouts prevent attaining the specified notch radius.

APPENDIX II

DECARBURIZATION AND INCLUSION STUDIES

Decarburization of thin sheet is difficult to avoid either in the mill or during heat treatment. Its effect depends on the carbon gradient produced through the sheet thickness. While a low-carbon ductile skin may lower the yield strength it also may increase the resistance to brittle fracture. On the other hand, loss of appreciable carbon throughout the sheet results in a significant loss in both tensile and yield strength.

In order to determine the carbon gradient in the sheet rather elaborate experimental techniques must be employed. The present results relate primarily to average carbon contents.

Referring to Table I, the carbon content of the ferritic alloys is given at the one quarter width position for the as-received and as-heat-treated sheet. Comparing the melt analysis with the as-received sheet carbon content, it is noted that some carbon loss occurred for SAE 4340 and AMS 6434 CEVM. Small carbon losses (two points) apparently also occurred during the heat treatment for SAE 4340, AMS 6434 (0.095-in.), UHS 280, and 300 M. Metallographic examination, however, did not reveal any decarburization in the as-heat-treated sheets. The carbon determinations for SAE 4340 and AMS 6434 (0.095-in.) were repeated after milling 0.010 in. from each side of the heat-treated sheet. These results show a carbon content then equal to the as-received sheet, indicating surface decarburization during heat treatment.

The above results may explain the low smooth strengths observed for SAE 4340 but cannot explain the lower yield strengths in the 0.063-in. as compared with 0.095-in. AMS 6434.

Inclusion ratings are given in Table II. Results were available for all alloys except 17-7 PH. The authors have no way of evaluating the meaning of these ratings either in terms of generally expected results for sheet alloys, or definitely establishing their influence on the reported strength values. It would appear that consumable electrode vacuum-melted AMS 6434 is cleaner than the corresponding air-melted alloy. This may in part be associated with the improved notch strength of the vacuum melted material. Also it is noted that both AISI type 301 and AISI type 304 L possess a comparatively high number of sulfide and silicate type inclusions and these may contribute to their poor transverse notch strength.

REFERENCES

(1) G. Sachs, "Survey of Low Alloy Aircraft Steels Heat Treated to High Strength Levels, Part 3—Failure Cases," *Technical Report No. 53-254*, Wright Air Development Center, Part 3, July 1954.

(2) G. Sachs, J. D. Lubahn, and L. J. Ebert, "Notch Bar Tensile Test Characteristics of Heat Treated Low Alloy Steels," *Transactions*, Am. Soc. Metals, Vol. 33, p. 340 (1944).

(3) G. Sachs, J. D. Lubahn, and L. J. Ebert, "The Effects of Notches of Varying Depth on the Strength of Heat-Treated Low-Alloy Steels," *Transactions*, Am. Soc. Metals, Vol. 35, p. 517 (1945).

(4) G. Sachs and J. D. Lubahn, "Effects of Notching on Strained Metals," *The Iron Age*, Oct. 8, 1942, p. 31; Oct. 15, p. 48.

(5) G. R. Irwin, J. A. Kies, and H. L. Smith, "Fracture Strengths Relative to Onset and Arrest of Crack Propagation," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 658 (1958).

(6) G. R. Irwin, J. A. Kies, and H. L. Smith, "Fracture Strengths Relative to Onset and Arrest of Crack Propagation," *NRL Report 522*, Naval Research Lab., Nov. 20, 1958.

(7) A. S. Pellini, G. Sandoz, and H. F. Bishop, "Notch Ductility of Nodular Iron," *Transactions*, Am. Soc. Metals, Vol. 46, p. 418 (1954).

(8) J. E. Srawley and C. D. Beachem, "Crack Propagation Tests of High Strength Sheet Steels Using Small Specimens," *NRL Report 5127*, Naval Research Lab., April 9, 1958.

(9) W. Buchmann, "Deformationless Breaks of Bolts of Cr-Ni-Mo Steel and Their Cause," *Mitteilungen Vereinigung Grosskesselbesitzer*, No. 65, p. 393 (1937).

(10) W. Kuntze, "The Practical Procedure for Notch Bar Tensile Tests," *Metallwirtschaft*, Vol. 11, p. 179 (1932).

(11) K. Richard, "The Strength Behavior of Low Alloy Heat Resisting Steels and the Tendency Toward Brittle Breaks," *Archiv für Metallkunde*, Vol. 3, p. 151 (1949).

(12) G. Sachs, L. J. Ebert, and W. F. Brown, Jr., "Comparison of Various Structural Alloy Steels by Means of the Static Notch Bar Tensile Test," *Metals Technology*, Technical Paper 2110; *Transactions*, Am. Inst. Mining and Metallurgical Engrs., p. 172 (1947).

(13) P. P. Puzak and J. Stoop, "Simple Laboratory Bulge Tests for Sheet Metal Fracture Transition Studies and for Weldment Performance Evaluation," *NRL Report 5347*, Naval Research Lab., 1959.

(14) G. Sachs, G. S. Sangdahl, and W. F. Brown, Jr., "New Notes on High Strength Heat Treated Steels," *The Iron Age*, Nov. 23 to 30, 1950.

(15) G. Sachs, J. G. Sessler, R. F. Pray, and T. H. Yeh, "Basic Relations for Notched Cylindrical and Prismatic Tension Test Specimens," Am. Soc. Mechanical Engrs. (*to be published*).

(16) G. Sachs and E. P. Klier; "Survey of Low Alloy Steels Heat Treated to High Strength Levels, Part 5—Mechanical Properties in the Presence of Stress Concentrations," *Technical Report No. 53-234*, Wright Air Development Center, Jan. 1954.

(17) E. P. Klier, B. B. Muvdi, and G. Sachs, "The Static Properties of Several High Strength Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 715 (1957).

(18) M. H. Jones and W. F. Brown, Jr., "An Axial Loading Creep Machine," ASTM BULLETIN, No. 211, Jan. 1956, p. 53 (TP 25).

(19) G. Sachs, "Survey of Low Alloy Aircraft Steels Heat Treated to High Strength Levels, Part 4—High Strength Steels and Their General Static Properties," *Technical Report No. 53-254*, Wright Air Development Center, Part 4, Dec. 1953.

(20) A. G. Alten and P. Payson, "The Effect of Silicon on the Tempering of Martensite," *Transactions*, Am. Soc. Metals, Vol. 44, p. 775 (1952).

(21) J. W. Sands, "Development of a Steel for the 280,000 to 300,000 psi Tensile Strength Bracket," Symposium on Ultra High Strength Steels in Aircraft Applications, *SP 120*, Soc. Automotive Engrs., p. 35 (1953).

(22) C. H. Shih, B. B. Averbach, and M. Cohen, "Some Effects of Silicon on the Mechanical Properties of High Strength Steels," *Transactions*, Am. Soc. Metals, Vol. 48, p. 86 (1956).

(23) E. J. Whittenberger and E. R. Rosenow, "Influence of Alloy Elements on the Impact Transition Behavior of 12 per cent Chromium Steels Aged at 900 F," *Transactions*, Am. Soc. Metals, Vol. 48, p. 391 (1956).

(24) W. C. Hagel, Discussion to E. J. Whittenberger and E. R. Rosenow's paper, *Ibid.* p. 414.

(25) G. Bandel and W. Tofaute, "The Brittleness of High Alloyed Chromium Steels in the Temperature Range of 500 C," *Archiv. für das Eisenhüttenwesen*, Vol. 15, Jan. 1942, p. 307.

(26) G. Sachs, L. J. Ebert, and W. F. Brown, Jr., "Notch-Tensile Characteristics of Partially Austempered Low Alloy Steels," *Metals Technology*, Technical Paper 2321, Feb. 1948.

(27) V. N. Krivobok, "Properties of Austenitic Stainless Steels at Low Temperatures." *NBS Circular No. 520*, Nat. Bureau of Standards, (1952).

(28) Heat Treatment and Physical Properties of the Austenitic Chromium-Nickel Stainless Steels—Nickel Alloy Steels," International Nickel Co., Section 7, Data sheet A, New York, N. Y. 1947.

(29) J. E. Srawley and C. D. Beachem, "Crack Propagation Tests of Some High Strength Sheet Steels." *NRL Report 5263*, Naval Research Lab., Jan. 10, 1959.

(30) J. L. Burns, T. L. Moore, and R. S. Archer, "Quantitative Hardenability," *Transactions*, Am. Soc. Metals, Vol. 26, p. 1 (1938).

(31) L. J. Klingler, W. J. Barnett, R. P. Frohmberg, and A. R. Troiano, "Embrittlement of Alloy Steels at High Strength Levels," *Transactions*, Am. Soc. Metals, Vol. 46, p. 1557 (1954).

DISCUSSION

Mr. J. E. Srawley[1] *(presented in written form)*.—Our approach to the problem of evaluating high-strength sheet materials for heavily loaded applications, while somewhat different in techniques and interpretation, has much in common with that of the authors. We would first like to endorse and emphasize their statement that a high yield strength is desirable only if coupled with satisfactory insensitivity to notches and adventitious cracks; otherwise brittle failure may occur at nominal stresses that are well below the yield strength. Surprisingly, this statement is not as universally accepted as it might be.

The load that can be sustained by a structure will depend upon the intensity of suitably oriented stress-concentrating flaws that happen to occur in that structure, and as the nominal stress level increases (while remaining less than the yield strength of the material) the critical flaw intensity becomes smaller. If it should become less than can confidently be detected by inspection methods applied to the structure (as it may with the materials of very high strength that we are considering), then it will be only a matter of chance whether or not the load can be brought up to the required level before fracture occurs.

The yield strength of a given material may be varied by appropriate treatments within a characteristic range. It seems to us that there is no good reason to use a material at such a level of strength that, for a given probable intensity of flaw, the nominal fracture stress will be less than the yield strength. By appropriate modification of the treatment, the yield strength can be lowered and the fracture stress for the given flaw intensity increased until the two coincide at a value greater than the fracture stress in the original condition. Thus the determination of the intensity of flaw that may escape inspection procedures becomes a matter of great importance. Unfortunately there is very little accurate information to be obtained on this, and thus any attempt to design or select materials quantitatively on the basis of the intensity of expected flaws is severely handicapped. However, if the intensity of the notch or crack used in the laboratory specimens is larger than the equivalent of what may confidently be expected to be discovered in the inspection of a structure, then the specimen will be more sensitive than the structure; thus a test criterion can be established that will at least reject all dangerous materials although it may also reject some that would not be dangerous in the structure. At the present time it is not even known whether the various laboratory specimens that are in use meet this requirement; if they do not, then there is a great need for improved inspection procedures. In this situation the best that can be done is to establish arbitrary criteria which will place materials in an order of merit with respect to their ability to accommodate flaws. Empirical correlation of these criteria with the behavior of actual structures will then be required before the tests can be used for material selection and quality control purposes.

---

[1] Metallurgy Division, U. S. Naval Research Laboratory, Washington, D. C.

In our tests we use a specimen that is different from that of the authors in that, instead of notching the edges, we provide a central transverse slot ending in cracks, which were formed in our early work by hydrogen embrittlement and slow straining but are now formed by fatigue stressing **(1, 2)**.[2] We believe that this type of specimen is more stable than the edge-notched type, extension of the crack at one end tending to increase the stress concentration at the other end. Furthermore, in the usual type of pressure vessel fracture, the crack propagates in two opposite directions, as in the center-cracked tension specimen.

We have studied both the net fracture stress (the equivalent of the authors' notch strength) and the appearance of the fracture, as a function of testing temperature, for a number of high-strength materials. We find that fracture appearance can be expressed quantitatively as the percentage of the thickness of the specimen occupied by the oblique, shear borders (as contrasted with the central band of fracture lying, on the average, in a plane normal to the applied load). This "per cent shear" suffers an abrupt increase with testing temperature from a low value (considerably less than 50 per cent) to a value of 100 per cent over a small range of temperature, in some cases less than 10 F. We refer to the lowest temperature at which a specimen would exhibit 100 per cent shear fracture as the fracture-appearance transition temperature (FATT). The net fracture stress suffers a similar change, and in this case we refer to the temperature at which it is equal to the yield strength as the net-fracture-stress transition temperature (NFSTT). Since the net fracture stress at any particular temperature is a function of both specimen dimensions and notch or crack intensity, there is no particular reason why the NFSTT should coincide with the FATT. Actually the two are often close and for specimens 0.5 in. wide the former is usually somewhat lower than the latter.

The FATT is believed to be quite insensitive to specimen dimensions except the thickness, which is a basic variable in the fracture of sheets, and to notch intensity beyond a certain point. This is one reason why we have tentatively selected the FATT as our criterion of relative brittleness. A complementary reason is that fracture appearance provides a unique means of directly comparing laboratory tests with service or proof testing failures—it is a feature exhibited by all fractures. We have suggested that a necessary condition, though not necessarily a sufficient one, for the suitability of a given material for a particular application is that the FATT for the thickness to be used should be lower than the lowest temperature at which the application is to be operated. We feel that in making the comparisons between data from our respective tests it would have been valuable if the authors had provided measurements of the per cent shear fracture exhibited by their specimens.

We have so far evaluated about 40 different combinations of material composition, condition (heat treatment), and thickness, and of these not one with a room temperature yield strength appreciably greater than 200,000 psi has had a FATT unambiguously lower than room temperature. This finding is in reasonable agreement with the authors' and also with that of other investigators working in the field. It is apparent that considerable work has yet to be done on the development of high-strength steels that are adequately insensitive to stress-concentrating defects.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this discussion.

One factor that needs to be emphasized is the effect of thickness. For a given material in a given condition the FATT increases markedly with thickness. For two steels which were investigated, the magnitude of the effect was about 70 F per 0.01 in. in one case and 40 F per 0.01 in. in the other case. The thicknesses ranged between 0.01 and 0.04 in. in the former and 0.043 and 0.10 in. in the latter material. A similar effect is found for any other reliable indicator of brittleness, as is to be expected from theoretical considerations. Thus it is of paramount importance that it should not be assumed that a material that has satisfactory properties in one particular thickness will also be suitable in a greater thickness —it is necessary to establish the suitability for the greatest thickness that is to be used. As a corollary to this, tests with scale-model vessels in which the wall thickness is scaled down proportionately may well be misleading as to the behavior of the full size vessel unless supplemented by an experimental determination of the magnitude of the thickness effect.

In this discussion we have concentrated upon the relatively narrow issue of the fracture toughness of the sheet materials themselves and have largely disregarded the factors in the construction of the vessels which have been responsible for most of the failures to date. Notable among these is the welding, and we believe that separate and extensive investigations of the various factors associated with welding is of vital importance. Either certain critical welds must be eliminated by resort to alternative fabrication procedures, or welding procedures must be developed which will ensure greater toughness in the vicinity of the welds than elsewhere. We have touched upon inspection which is intimately and inextricably involved in the whole problem. Nevertheless, if further advances are to be made without radical changes in methods of construction, the search for materials with higher strength and adequate toughness must continue, and it is necessary to develop suitable means of evaluating such materials which are less expensive than testing pressure vessels to destruction.

MR. JOHN G. SESSLER[3] (*presented in written form*).—This paper contributes a wealth of information on the behavior of high-strength steel sheet alloys in the presence of high-stress raisers.

We agree with the authors' opinion that the sharp edge-notch sheet tension specimen is quite suitable for purposes of high-strength sheet alloy development and evaluation. It has been shown that this specimen is extremely sensitive to known embrittling factors, such as those produced by heat treatment, and will yield good reproducible test results.

In addition, we would like to point out another important advantage of the edge-notch sheet specimen, namely, that a wide range of elastic stress concentration factors may be introduced into this specimen simply by varying the geometry of the notch.

At Syracuse, we are presently conducting a program, sponsored by the Bureau of Aeronautics, which includes the heat-treated titanium sheet alloys Department of Defense (DOD) plus some of the high-strength steel alloys discussed by the authors.

In the authors' program, a single notch geometry (very sharp notch) was used to study the effects of heat treatment, or conditions of cold work, that produce a wide range of strength levels for each alloy investigated. In our program, we are investigating a single strength level and varying the notch geometry (and thus varying K_t). Our primary objective has been to study the behavior of high-strength sheet over a

---

[3] Syracuse University, Research Institute, Syracuse, N. Y.

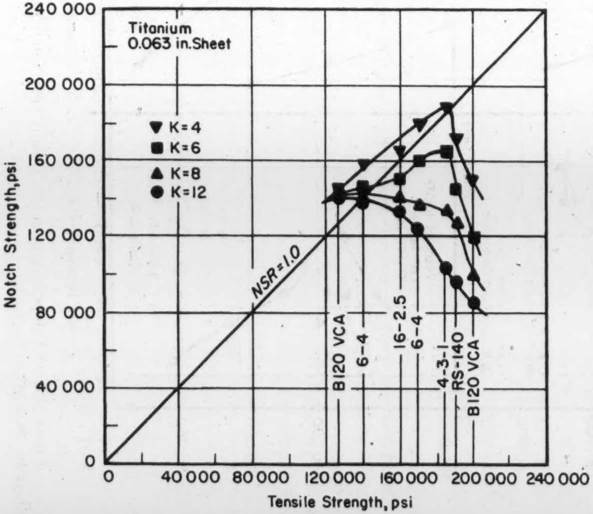


FIG. 22.—Effect of Tensile Strength Level on the Notch Strength of Various Titanium Sheet Alloys for Several *K* Factors.

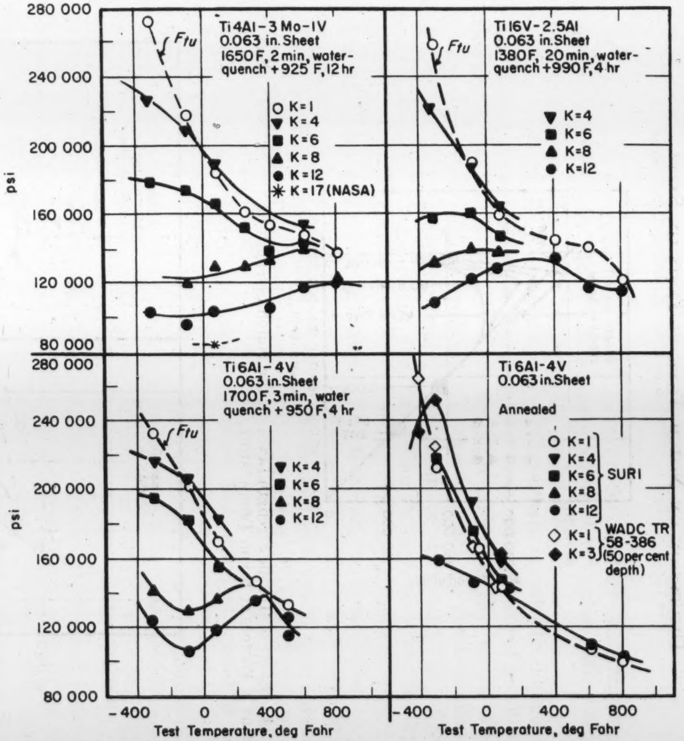


FIG. 23.—Effect of Test Temperature on the Tensile and Notch Tensile Strength of Various Titanium Sheet Alloys for Several *K* Factors.

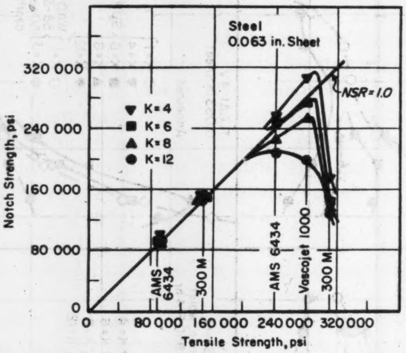


FIG. 24.—Effect of Tensile Strength Level on the Notch Strength of Several Steel Sheet Alloys for Various *K* Factors at Room Temperature.

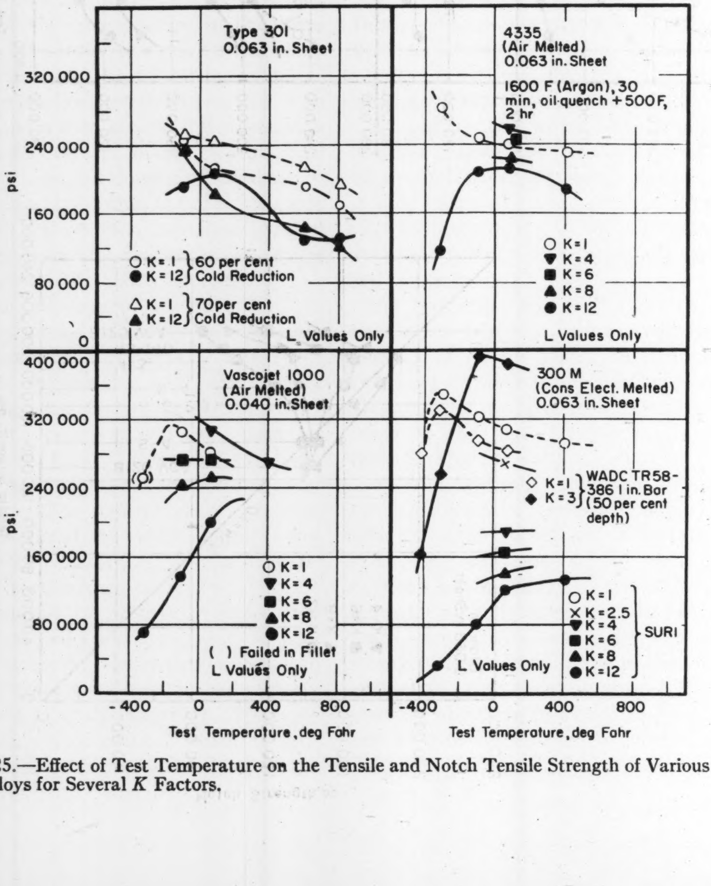


FIG. 25.—Effect of Test Temperature on the Tensile and Notch Tensile Strength of Various Steel Sheet Alloys for Several *K* Factors.

range of stress concentrations from very mild notches to the very sharp notch used by the authors. The effect of test temperature and specimen orientation have also been studied for a few representative alloys.

The accompanying figures present typical test data obtained in our study, which we believe may supplement the data presented by the authors.

Figure 22 shows the effect of stress concentrations on the notch tensile strength of several titanium sheet alloys at room temperature. The effect of test temperature is shown in Fig. 23. The same type of data is given for several high-strength steels in Figs. 24 and 25, respectively.

To date, most of the data have been acquired using a single sheet thickness and a single width specimen. Investigation of gage and width (section size) have been recently initiated.

MR. E. P. KLIER[4] (*presented in written form*).—Perhaps the most striking conclusion that is to be drawn from the authors' work is that at this time there are no steels available today that are notch-tough at yield strengths above 200,000 psi. Such yield strengths can lead to potential yield strengths of significantly higher values under combined stresses, but limiting values less than about 250,000 psi are clearly indicated at this time.

It is interesting to note that several of the steels have been somewhat irregular in heat treatment, according to the authors' Fig. 12, notably SAE 4340, AMS 6434, and possibly UHS 280 steels. We wish to point out that we have recently been able to show that this behavior is due to transformation characteristics in these steels which have heretofore not been recognized. Initial slow cooling from the normal austenitizing temperature to an intermediate temperature followed by quenching tends to suppress the low hardness effect observed by the authors.

It is perhaps in order to emphasize one point concerning the use of notch testing. It is probable that the testing of metals in the presence of notches allows the collection of data that are effectively available through no other testing procedure. The significance of notch tests, therefore, remains regardless of their correlation in a simple way with the performance of structures. As a corollary to this, if the performance of structures can be faithfully predicted from the result of a notch test, this test should be used as needed. On the other hand if a simpler test can provide the information required it should not be overlooked because it contains no notch. What is of concern here is the nature of the embrittling agent. For example, several factors have been advanced in the paper as embrittling agents; we have in mind especially local nil ductility; while one of the most serious agents of brittle fracture, namely, welding, is mentioned in passing. Yet it is not at all evident how these two conditions are amenable to notch testing. For satisfactory service performance these conditions must be subject to control in an unequivocal manner.

It is suggested at this time, therefore, that notch testing especially aimed at developing and fully evaluating potential new high yield strength materials, to ensure a reference line for material notch toughness properties, be prosecuted. It is suggested, however, that such testing alone at its present state of development cannot give a satisfactory picture of the properties modifications particularly due to welding, and that as a consequence tests which allow weld properties evaluation should be developed.

MR. J. C. HAMAKER, JR.[5] (*presented*

---

[4] Research Professor, Catholic University, Washington, D. C.

[5] Director of Research and Metallurgical Engineering, Vanadium-Alloys Steel Co., Latrobe, Pa.

in written form).—The authors should be commended for their comprehensive approach to this problem of notch or crack propagation in ultra-high-strength sheet material. The application of one test method to a representative group of steels over the entire range of their strength capabilities provides notch property comparisons that have not heretofore been available.

Figure 21 of the authors' paper is particularly informative and indicates that all materials are quite similar in their loss of notch strength (or increased susceptibility to crack propagation) as the heat-treated or cold-worked strength level is raised. It is noteworthy that the relative positions of the steels in this figure are changed considerably when transverse rather than longitudinal properties are plotted. Under these conditions, the stainless steels AISI 301 and PH 15-7 Mo reverse their positions relative to the other materials. Furthermore, in the air-melted condition, AMS 6434 steel practically coincides with the other materials on both longitudinal and transverse tests. Thus, from an over-all evaluation, the only significant improvement appears to arise from the consumable electrode vacuum melting of AMS 6434 steel. This would suggest that priority should be given to the effect of vacuum melting on all of the materials before definite conclusions are made regarding the relative merits of the various alloy systems and strengthening mechanisms.

Another factor that should be considered in the interpretation of data of this type is the maximum strength capability of each steel investigated. For example, if the 250,000 psi tensile strength level is being considered for a given design, the carbon content of the given alloy system might well be adjusted to provide just that strength capability and no higher. The 0.35 per cent carbon AMS 6434 steel, having a strength capability of only 250,000 psi, could be expected to have an inherent ductility advantage over other materials capable of achieving 300,000 psi. A recently completed investigation on the effect of carbon content in 5 per cent chromium steels indicates a broad range of strength and ductility combinations available in this system. For the 250,000 psi ultimate tensile strength level, a 0.22 per cent carbon modification can be selected having appreciably higher ductility than the 0.40 per cent carbon Vascojet 1000 tested in this investigation. For strength levels above the latter's 300,000 psi capability, a 0.50 per cent carbon modification achieves the 325,000 to 330,000 psi range with somewhat lower ductility. Thus, to obtain the most accurate comparison of relative properties, it might be advisable in the future to plot summary curves like those of the authors' Fig. 21 only by groups of steels having similar maximum strength capability.

The authors' comments in the section "Practical Significance of Results" should be particularly helpful to designers. Field reports of burst stresses exceeding 300,000 psi in welded pressure vessels of 5 per cent chromium steel, at strength levels where relatively low notch strengths are indicated, lends confirmation to the authors' suggestion that proper care in fabrication and heat treatment may offset this problem. Also, from a practical viewpoint, the marked similarity in notch behavior of all materials at increasing strength levels would suggest that properties other than notch sensitivity may become controlling factors in material selection. For example, heat treatment by air hardening and tempering at high temperatures may significantly improve performance of actual pressure vessels by minimizing distortion and residual stress, and completely removing the retained austenite that can tend to transform to brittle martensite on proof testing for potentially reduced ductility in service.

MR. W. J. RUPRECHT[6] (*presented in written form*).—First the authors are to be complimented for the presentation of a thorough and exacting treatment of tension testing. It is always a pleasure to observe data which have been developed with: (1) the major testing variables accurately defined and controlled, (2) the test specimens precisely described, and (3) the test equipment noted.

We have observed a general trend in the United States, occurring over a number of years, that would indicate a general "sloppiness" due either to ignorance or possibly a relaxed attitude on the part of technical people to exacting methods of test. This attitude has resulted in a state of general confusion in the ultra high-strength steel area. We know of instances in both Government and private industry where data have been presented without regard to test specimen shape and size or test method. Intercomparison of many sources of data has thus become impractical and has resulted in much wasted effort.

We wish to support the authors' work with additional data obtained using similar methods but developed by slightly different techniques in specimen fabrication and test conditions.

Two steels were investigated, namely, AMS 6434 (modified) and AISI E4340. Both steels were used in missile work conducted within our company. The notched specimen geometry used was the same as the authors. The smooth tension specimens corresponded to the standard 0.500-in. rectangular tension test specimen using a 2-in. gage length.[7] The specimens flat surfaces were allowed to remain in the as-rolled condition. All specimens were heat treated after finished machining. Heating was accomplished in neutral salt. The detailed metallurgical conditions of the test materials is known, but will not be discussed. The AMS 6434 (modified) steel was investigated using two thicknesses, 0.095 in. and 0.220 in. One thickness 0.095-in. AISI E4340 steel was used.

All specimens were tested in a Universal testing machine. The smooth tension specimens were gripped using a friction chuck. The notched tension specimens were tested using special pin grips similar to those used by the authors. Smooth tensile yield strength was determined using an averaging separable pin-type PS-5M microformer extensometer to determine strain. Autographic curves were generated on a model MA-1 microformer-type Baldwin stress-strain recorder. The rate of loading was 2000 lb per min for the 0.095-in. specimens and 4000 lb per min for the 0.220-in. specimens. Breaking load was taken from the machine dial reading. The smooth tension test specimens were tested in accordance with Federal Test Method Standard No. 151. A minimum of three specimens were tested under each condition.

The results are presented in Fig. 26. Circular points are the result of single tempering treatments; triangular points represent double tempering cycles. For the AMS 6434 (modified) steel the austenitizing temperature was 1625 F and austenitizing time was 30 min at temperature. The specimens were then quenched in salt at 390 to 400 F followed by an air cool to ambient. The holding time in the quenching medium was 5 min for the 0.095-in. specimens and 10 min for 0.220-in. specimens. The AISI E4340 steel was austenitized in salt at 1500 F holding 20 min at temperature. Specimens were then quenched in salt at 390 to 400 F

---

[6] Supervisor, Metallurgical Research, American Car and Foundry, Berwick, Pa.

[7] The net fracture stress is computed by dividing the maximum load by the uncracked area at the start of fast fracture. This area may be determined using the Irwin ink stain technique.

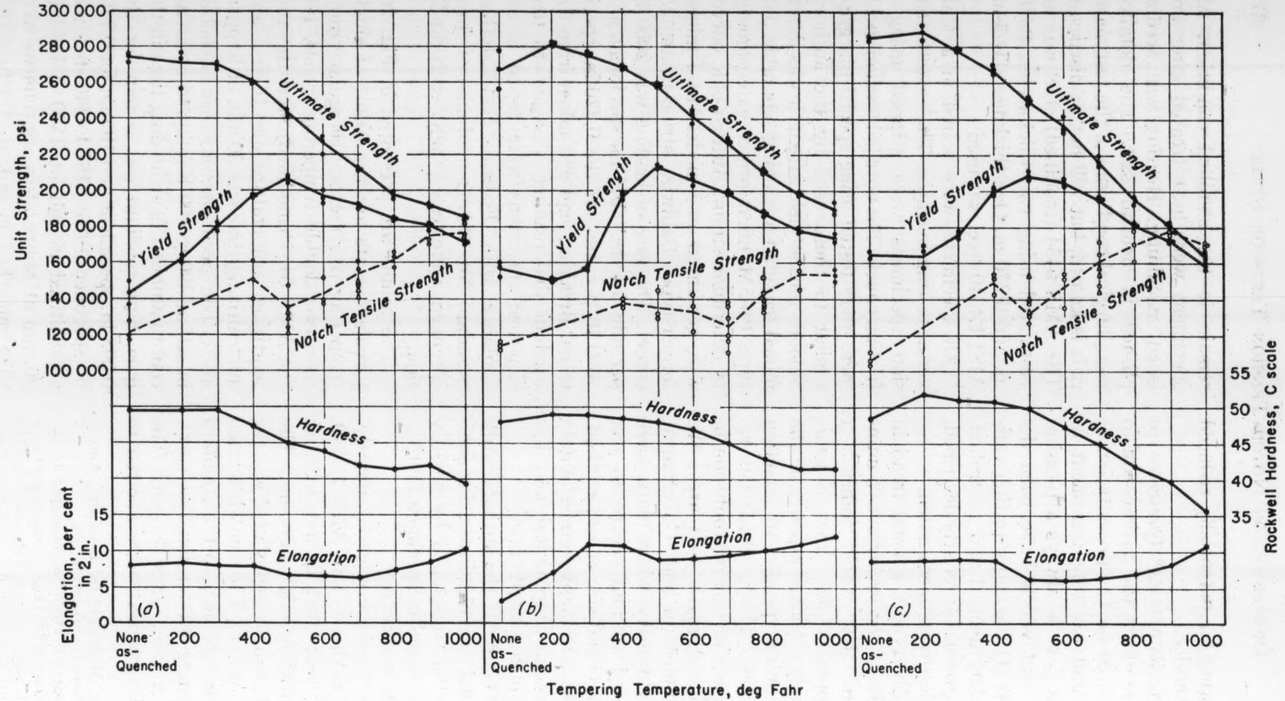


FIG. 26.—Mechanical Properties of AMS 6434 and AISI 4340 Steels.
(*a*) 0.095-in. Thick AMS 6434 (Modified) Steel.
(*b*) 0.220-in. Thick AMS 6434 (Modified) Steel.
(*c*) 0.095-in. Thick AISI 4340 Steel.

for 5 min followed by an air cool to ambient. The specific heat treatments were dictated by the heat-treating practice used in fabricating 54-in. diameter solid propellant missile cases.

The data presented represent an initial effort to use the notched tension specimen in an investigation of 54-in. diameter missile case failures. Prior to this investigation a number of AMS 6434 (modified) case failures had been experienced. The cases were heat treated to develop optimum yield strength, tempering temperature 500 F. Failure origin areas were intergranular cleavage-type fractures. As a result of the notched tensile data, the tempering temperature was raised to 900 F. No additional failures were experienced. Fabrication techniques were judged comparable for both the failed and successful cases. Therefore the notched tensile strength test is believed to approach a condition of preexisting microcracking known to have been present in a number of the failed cases and to accurately indicate the notch brittle area of the respective tempering curves.

Subsequent work and careful attention to the notch geometry has allowed triplicate notched tensile values with a maximum 5000 to 8000 psi variation in any given set of conditions. This value should be judged in light of the fact that the certified accuracy of the testing machine is ±1 per cent and in the range considered would contribute 3000 to 4000 psi variation to the over-all results.

MESSRS. G. B. ESPEY, M. H. JONES, AND W. F. BROWN, JR. (*authors' closure*). —The authors are gratified by the thorough discussion and evident interest in this investigation.

Mr. Srawley has presented a most interesting and informative discussion which very well summarizes his views on the subject of fracture testing. We are in general agreement with the substance of his philosophy and have only one comment. He has concentrated considerable attention on the fracture appearance, and the authors agree these data are very valuable for the reasons stated. However, the interpretation of fracture appearance may be somewhat complicated by what has been called "low energy shear failures." These are ductile appearing fractures which behave in other respects much like the normal cleavage type. Some recent tests which illustrate this point are shown in Fig. 27 for 0.40 per cent carbon 300M steel. This plot is similar to those shown in the paper, except that the per cent shear and the net fracture stress,[7] σ_x, has been added. It will be noted that 100 per cent shear characterizes the material tempered at 1150 F and as expected at this temperature, both the net fracture stress and the notch strength exceed the yield strength. However, 100 per cent shear also characterizes tempering temperatures of 700 F and lower and in this range both the net fracture stress and the notch strength are well below the yield strength.

Mr. Sessler has pointed out that the edge notch tensile specimen is well suited to the investigation of notch sharpness effects. These effects are very important to the designer because not all stress concentrations result from crack-like flaws. Frequently they arise from unavoidable section size changes such as threads or fillets. Neither the sharp notch nor the center crack specimen can even qualitatively predict the tendency to fracture at low average stress in the presence of such gross discontinuities. The problem here is probably one of crack initiation under the particular strain gradients produced by the notch geometries involved. This is a problem for which we have very little fundamental information. The data presented by Mr. Sessler are quite interesting to us in several respects. First, they show the notch radius effect may be unexpectedly complex. For example, in Mr. Sessler's Fig. 25 the room temperature notch strength of 300M decreases very rapidly in a narrow range of stress concentration factors (between 2.5 and 4). This behavior reminds one of the transition in notch properties over a narrow range of test temperature as emphasized by Srawley. Second, as shown in Sessler's Fig. 23, a reduction in the edge notch radius from 0.002 in. ($K = 12$) to values less than 0.001 in. ($K \sim 17$) appreciably reduces the notch strength of 4-3-1 titanium.

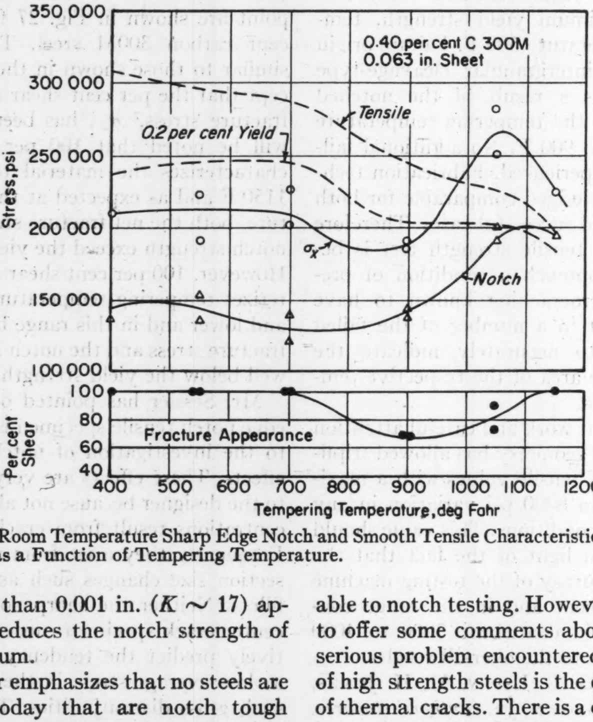


FIG. 27.—Room Temperature Sharp Edge Notch and Smooth Tensile Characteristics and Fracture Appearance as a Function of Tempering Temperature.

Mr. Klier emphasizes that no steels are available today that are notch tough above about 200,000, psi tensile yield strength. The authors wish to point out that this statement applies specifically to the particular geometry tested. The specimen represents a rather severe flaw condition in the alloy and it is not strictly correct to assign limits to the usable yield strength on the basis of this test alone.

Mr. Klier feels that if certain of the low-alloy steels were heat treated in a different manner the hardnesses for the low tempers would be raised. This is entirely possible and further work is certainly desirable in this direction. However, the treatments used in this investigation were selected to represent current practice in the missile industry. The authors agree with Mr. Klier that it is not yet clear how welding embrittlement is amenable to notch testing. However, they wish to offer some comments about this. One serious problem encountered in welding of high strength steels is the development of thermal cracks. There is a continuously increasing body of evidence which indicates some of the metallurgical factors active in promoting thermal cracking are responsible also for reduced notch toughness in the parent metal. Outstanding among these factors is the deleterious effect of carbon in excess of that required to give the desired strength level. In addition as shown, by Cottrell and Wilkinson (3), the tendency to thermal cracking as well as the impact transition temperature increase with increase in the phosphorus and sulfur content. It, therefore, might be expected that an improvement in notch toughness of the parent metal would often lead to improved toughness in the weldment. If the welding variables are to be studied directly, the authors suggest the use of a notch test. Thus, the notch may be introduced at a series of chosen locations about the weld, each location requiring a separate specimen cut from a welded plate. This type of investigation was made a number of years ago by one of the authors (4). The material was annealed firebox steel, and quite ductile. However, the techniques used clearly outlined regions of embrittlement and the effects of welding variables or heat treatment on these zones. The same techniques should be applicable to welded high-strength sheet.

The authors agree in general with the thoughts expressed by Mr. Hamaker. However, they wish to make specific comments on a number of his points. Referring to Fig. 21 in the authors' paper, it is true that if transverse properties were plotted a different order of the curves would arise. The difference between longitudinal and transverse properties is brought out in Figs. 19 and 20 and in Table IV. The authors chose the longitudinal direction for Fig. 21 because it frequently exhibits lower notch sensitivity and may be more nearly representative of the alloys potential. Furthermore, in some designs, the sheet can be oriented with the largest stress in the longitudinal direction. Mr. Hamaker considers that practically no difference exists between the various alloys in Fig. 21 when air melt ASM 6434 is substituted for the vacuum melt material. It should be noted that data for air melted AMS 6434 is reported for both 0.063 in. and 0.095-in. thicknesses (see Fig. 18). Mr. Hamaker's statement applies best to the 0.095-in. thickness. Air melt 0.063-in. AMS 6434 appears superior in notch toughness to the other 0.063-in. thick ferritic steels at the 250,000 psi tensile strength level. Mr. Hamaker prefers to compare materials by groups having similar maximum strength capabilities. The authors agree that a comparison on the basis of maximum strength capability would be valuable, particularly if one were describing the effect of some variable which does not in itself appreciably affect the smooth tensile strength, such as melting practice or phosphorus and sulfur content. However, the designer asks a different question, namely, of all the materials available, which one has the best notch properties at a given strength level. There is no *a priori* reason for assuming the material with the lowest maximum tensile strength will have the highest notch strength ratio (for example, compare PH 15-7 molybdenum and AMS 6434 in Fig. 21). Therefore, Fig. 21 serves a definite purpose. The authors agree with Mr. Hamaker that reducing the carbon content of tempered martensites to as low a value as is consistent with the desired strength is generally a valuable concept and this is spelled out in the paper. If this is done it may well be that some of the high bred steels will be equal in notch toughness to AMS 6434 at comparable strength levels. However, notch data on such low-carbon modifications are not generally available nor are these compositions readily available on today's market. Another point is that the notch properties of tempered martensites are not only a function of carbon content, but, also, most likely depend to some extent on the relative amounts of other alloying elements.

Mr. Ruprecht has presented very interesting data. It is gratifying to the authors that another laboratory has produced edge notches to the specified sharpness using a different machining technique. Mr. Ruprecht has made a systematic study of the effect of tempering temperature on the sharp edge notch strength of 0.095-in. thick AMS 6434 (modified) and SAE 4340 sheet steel. The same "optimum" tempering temperatures are defined by these data and by the authors' data in Figs. 2 and 3. It is particularly encouraging that application of these optimum heat treatments to large missile cases eliminated previous difficulties with brittle fracture. It is hoped that Mr. Ruprecht's experience will encourage others engaged in the development and application of high-strength materials to use laboratory tests containing very high stress concentrations. By doing so, a great savings in effort, time and money may be possible.

REFERENCES

(1) J. E. Srawley and C. D. Beachem, "Crack Propagation Tests of High Strength Sheet Steels Using Small Specimens," *NRL Report 5127*, April 1958.

(2) J. E. Srawley and C. D. Beachem, "Crack Propagation Tests of Some High-Strength Sheet Steels," *NRL Report 5263*, Jan. 1959.

(3) F. J. Wilkinson, C. L. M. Cottrell and H. V. Huxley, "Calculating the Hot Cracking Resistance of High Tensile Alloy Steel," *British Welding Journal*, Dec., 1958, p. 557.

(4) W. F. Brown, Jr., L. J. Ebert, and G. Sachs, "Distribution of Strength and Ductility in Welded Steel Plate as Revealed by the Static Notch Bar Tensile Test," *Welding Research Supplement*, Oct., 1947, p. 545S.

EXPERIMENTAL DETERMINATION OF ENERGY RELEASE RATE FOR NOTCH BENDING AND NOTCH TENSION*

BY J. D. LUBAHN[1]

SYNOPSIS

Values of energy release rate were determined experimentally, as a function of notch depth, for notch bending and notch tension; and by means of fracture tests the critical energy release rate was determined for two steels. The experimental values of energy release rate showed fair to good agreement with analytical values available from the literature, depending on notch depth and type of test. In terms of the Griffith-Irwin concept of a critical energy release rate, the effects of notch depth and specimen size could be accurately predicted if extensive plastic flow did not occur, but correlation of different types of tests by means of this concept were only fair.

A recent comparison (1)[2] between notched disk bursting tests and notched bend tests indicates that one type of test can be related to another in terms of the "critical energy release rate." This quantity was introduced by Irwin (2) in connection with the Griffith-Irwin theory, which is an outgrowth of Griffith's original theory (3) applying to glass. The Griffith-Irwin theory states that rapid crack extension will occur whenever a small increase in crack area would result in the release of at least as much elastically stored energy from the piece as the energy required to form the crack.

It can be shown that the energy release due to an increment of crack area increases as the square of the load. On the other hand, the energy required to form the crack is presumably independent of load. Because of the way in which the elastic energy release and energy absorption by crack formation depend on load, an increment of crack area will not result in as much energy release as energy absorption at low load, but *will* result in as much energy release as energy absorption at some higher load. This higher load is the critical load for fracture, according to the Griffith-Irwin theory, and the corresponding rate of release of elastically-stored energy, measured with respect to crack area, is the "critical energy release rate," $\mathcal{G}_c$. The critical energy release rate, according to the theory, may be regarded as a material property in much the same way that modulus of elasticity is considered as a material property.

To apply the Griffith-Irwin theory to a particular situation, it is necessary to know the value of the critical energy release rate $\mathcal{G}_c$ and how the energy release rate $\mathcal{G}$ depends on load or nominal stress (that is, the coefficient of the square of the nominal stress). The critical energy release rate must be determined experimentally by substituting a measured fracture stress into an expression for the energy release rate for the pertinent situation. Thus, in any case, an expression for energy release rate is needed for practical application of the theory, in terms of the square of the nominal stress and other dimensions of the part, for both the engineering application and the test specimen.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] General Electric Co., Knolls Atomic Power Laboratory, Schenectady, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

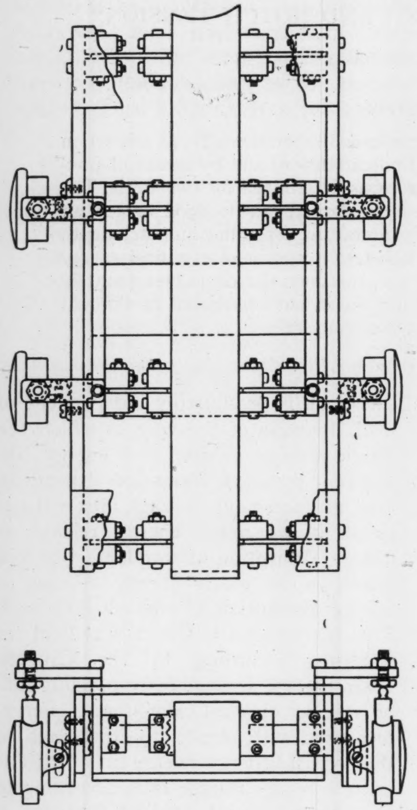


FIG. 1.—Deflection Gage Used in Bend Tests.

Analytically determined expressions for energy release rate are available in the literature for a few special cases, such as notch bending and notch disk bursting (1). Analytical solutions for energy release rate are very difficult mathematically for any but the most elementary physical situations. Consequently, it is desirable to be able to determine energy release rate experimentally. Irwin (4) has suggested a method of doing so, and this paper gives the result of applying Irwin's method to two cases—notch bending and notch tension (see Appendices III and IV).

EXPERIMENTAL METHOD

The bend specimen was made from a heat-treated nickel-molybdenum-vanadium steel forging[3] 2 in. square,[4] supported on $15\frac{1}{4}$-in. centers, and loaded on 6-in. centers. The supporting and loading knife edges had a $\frac{1}{2}$-in. radius. At midspan on the tension side, a slot was milled into the specimen. This slot was 16 mils wide and had a 60 deg angle at the bottom. By measuring the profile radius of

---

[3] This forging has been designated as the "Arizona rotor forging" in reference (7). The reference gives the composition, heat treatment, and properties of the steel. In the following, this material will be referred to as "material A."

[4] Conditions of plane strain (very wide beam) were desired. The following notch bend tests by S. Yukawa on a nickel-molybdenum-vanadium heat-treated steel indicate that the lowest strength of notched bend specimens has already been reached long before the ratio of width to gross beam depth reaches as large a value as unity.

Gross Beam Depth, in.	Net Beam Depth, in.	Beam Width, in.	Root Radius, mils	Nominal Strength, psi
4	3.2	$\frac{1}{8}$	3	128 500
4	3.2	$\frac{3}{8}$	5	74 500
4	3.2	1	3	61 400
4	3.2	1	10	70 200
4	3.2	4	10	94 500

the teeth on the cutter after the last cut, it could be established that the maximum root radius of the notch was 3 mils.

The method requires very accurate deflection measurements (see Appendix III). Deflection measurements at the loading points relative to the supports were made for notch depths of zero, 200, 400, 610, 800, and 1000 mils. For shallow notches (200 and 400 mils), the deflection measurements could be continued to a 20,000-lb load, thereby giving deflections of a magnitude that could be measured with some accuracy. For deeper notches, the load had to be limited to 10,000 lb to avoid fracture,[5] as discussed in Appendix I; but no loss of accuracy resulted because there is more deflection per unit load for the deeper notches.

Deflections were measured at the neutral axis by attaching two pairs of stiff measuring bars to the specimen with strips of spring steel, as shown in Figs. 1 and 2. One pair of measuring bars was attached to the specimen at the loading points and the other pair at the support points. The two bars of a pair were attached together to form a rigid "box." Thus, the relative motion of the two boxes was identically the same as the de-

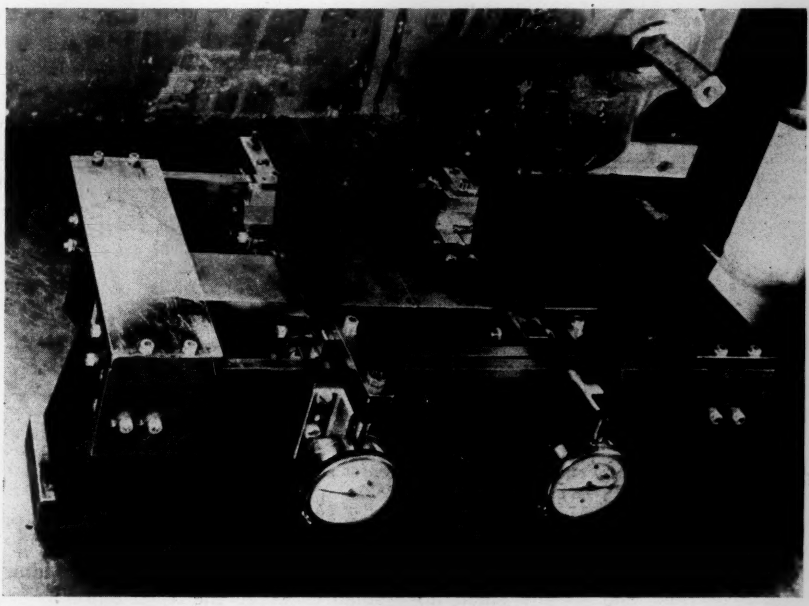


FIG. 2.—Deflection Gage Used in Bend Tests.

---

[5] The material for the tests was purposely selected for its fracture characteristics, namely, that fracture would occur in a 2-in. beam before extensive plastic flow. Thus, by avoiding fracture, extensive plastic flow is also automatically avoided. If a material had been selected which would suffer extensive plastic flow before fracture, such plastic flow would have to be avoided by limiting the maximum load.

flection at the load points. This relative motion was measured by four 0.0001-in. dial gages located at the four corners of the inner box.

The dial gages were calibrated for tooth profile error against a Pratt & Whitney supermicrometer. A typical calibration curve is shown in Fig. 3. However, the method of taking and analyzing the data, as described below, was such that it was unnecessary to apply corrections to the gage readings.

In determining the energy release rate, we are concerned only with the excess deflection of a notched specimen over that of the unnotched specimen. This extra deflection is small compared to the total deflection, and so any small absolute tooth profile errors in the total deflection would result in large relative errors in the excess deflection due to notching. The tooth profile error associated with the unnotched deflection was made to cancel out in calculating the excess deflection. This was done by setting the rack of the gage at the same location at zero load for both the notched and the unnotched tests. Consequently, the only error remaining was that associated with the excess deflection, and that was small enough to be disregarded (about 1 per cent).

To obtain the greatest accuracy in deflection values, it was found desirable to calculate the small differences between the actual load-deflection curve and a linear load-deflection curve of suitable slope, and average these differences. These differences will be called the "reduced deflections." Table I shows a typical calculation and Fig. 4 a typical result. All curves exhibited the characteristic initial hook shown by the empty circles in Fig. 4. This hook was due partly, but not entirely, to the characteristics of the calibration curve; after correction, the hook is still there but to a smaller degree. The hook appears pronounced when represented as in Fig. 4, but actually it constitutes an exceedingly small effect; in the usual representation (Fig. 5), the deviation from linearity is almost indistinguishable.

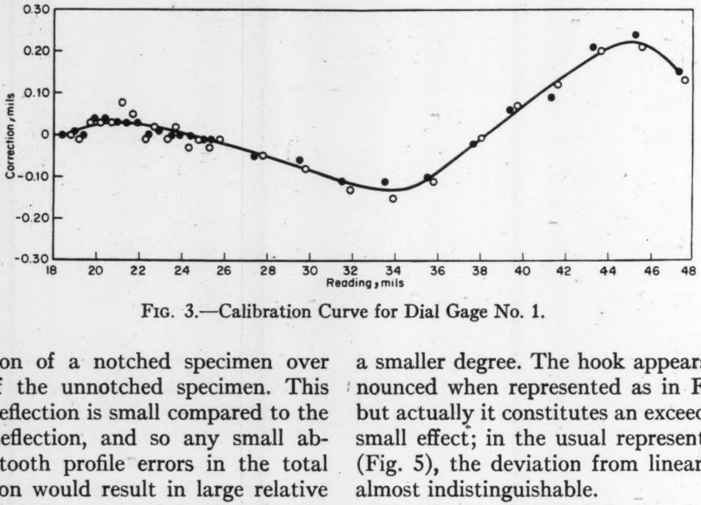


FIG. 3.—Calibration Curve for Dial Gage No. 1.

The final value of deflection for the test was obtained by adding the reduced deflection, as read from the faired curve, and the linear deflection. In the case of Fig. 4, for example, the deflection at 20,000 lb is:

$$(0.12 - 0.46)(10^{-3}) + (20{,}000)(1.25 \times 10^{-6}) = 24.66 \times 10^{-3}$$

and for 10,000 lb is:

$$(0.42 - 0.46)(10^{-3}) + (10{,}000)(1.25 \times 10^{-6}) = 12.46 \times 10^{-3}.$$

TABLE I.—CALCULATION OF AVERAGE REDUCED DEFLECTION FOR 200-MIL SLOT DEPTH.

Load, lb	Reading, mils	Linear Deflection, mils	Reduced Deflection, mils	Reading, mils	Linear Deflection, mils	Reduced Deflection, mils	Average Reduced Deflection, mils
	GAGE NO. 1			GAGE NO. 2			
0	19.86	19.0	0.86	70.58	69.6	0.98	...
1 000	21.02	20.3	0.72	71.81	70.9	0.91	...
2 000	22.35	21.6	0.75	73.17	72.2	0.97	...
3 000	23.58	22.9	0.68	74.46	73.5	0.96	...
4 000	24.87	24.2	0.67	75.72	74.8	0.92	...
5 000	26.19	25.5	0.69	76.98	76.1	0.88	...
6 000	27.46	26.8	0.66	78.24	77.4	0.84	...
7 000	28.65	28.1	0.55	79.48	78.7	0.78	...
8 000	30.02	29.4	0.62	80.73	80.0	0.73	...
9 000	31.30	30.7	0.60	81.93	81.3	0.63	...
10 000	32.63	32.0	0.63	83.27	82.6	0.67	...
11 000	33.82	33.3	0.52	84.42	83.9	0.52	...
12 000	35.12	34.6	0.52	85.62	85.2	0.42	...
13 000	36.34	35.9	0.44	86.88	86.5	0.38	...
14 000	37.58	37.2	0.38	88.18	87.8	0.38	...
15 000	38.68	38.5	0.18	89.33	89.1	0.23	...
16 000	40.03	39.8	0.23	90.62	90.4	0.22	...
17 000	41.28	41.1	0.18	91.79	91.7	0.09	...
18 000	42.58	42.4	0.18	93.13	93.0	0.13	...
19 000	43.71	43.7	0.01	94.32	94.3	0.02	...
20 000	45.02	45.0	0.02	95.61	95.6	0.01	...
	GAGE NO. 3			GAGE NO. 4			
0	19.38	18.9	0.48	20.23	19.2	1.03	0.84
1 000	20.67	20.2	0.47	21.48	20.5	0.98	0.76
2 000	22.07	21.5	0.57	22.90	21.8	1.10	0.85
3 000	23.42	22.8	0.62	24.23	23.1	1.13	0.85
4 000	24.72	24.1	0.62	25.40	24.4	1.00	0.80
5 000	26.03	25.4	0.63	26.66	25.7	0.96	0.79
6 000	27.27	26.7	0.57	27.87	27.0	0.87	0.74
7 000	28.54	28.0	0.54	29.03	28.3	0.73	0.65
8 000	29.84	29.3	0.54	30.28	29.6	0.68	0.64
9 000	31.06	30.6	0.46	31.52	30.9	0.62	0.58
10 000	32.41	31.9	0.51	32.88	32.2	0.68	0.62
11 000	33.58	33.2	0.38	34.03	33.5	0.53	0.49
12 000	34.82	34.5	0.32	35.28	34.8	0.48	0.44
13 000	36.06	35.8	0.26	36.52	36.1	0.42	0.38
14 000	37.31	37.1	0.21	37.88	37.4	0.48	0.36
15 000	38.50	38.4	0.10	39.02	38.7	0.32	0.21
16 000	39.76	39.7	0.06	40.25	40.0	0.25	0.19
17 000	40.98	41.0	−0.02	41.76	41.3	0.46	0.18
18 000	42.36	42.3	0.06	42.78	42.6	0.18	0.14
19 000	43.60	43.6	0.00	43.91	43.9	0.01	0.01
20 000	44.92	44.9	0.02	45.23	45.2	0.03	0.02

The deflections are summarized in Table II, which also shows the values of $\Delta\theta$ and K needed for calculating energy release rate. The quantity $\Delta\theta$ is defined as the angular deflection in excess of that for the unnotched beam, and given by:

$$\Delta\theta = \frac{\beta_N - \beta_U}{l}$$

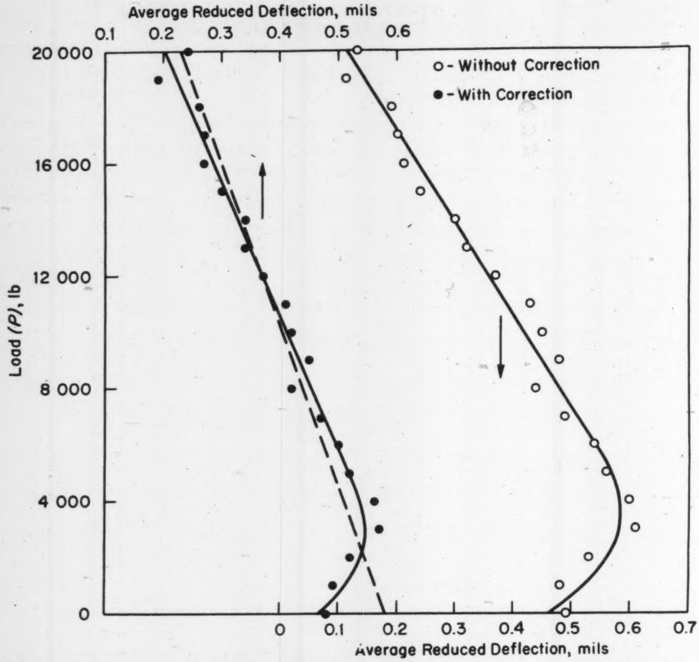


FIG. 4.—Typical Example of Load-Deflection Curve With and Without Correction for Gage Calibration. Deflections reduced by $1.25P \times 10^{-6}$. First test with zero notch depth.

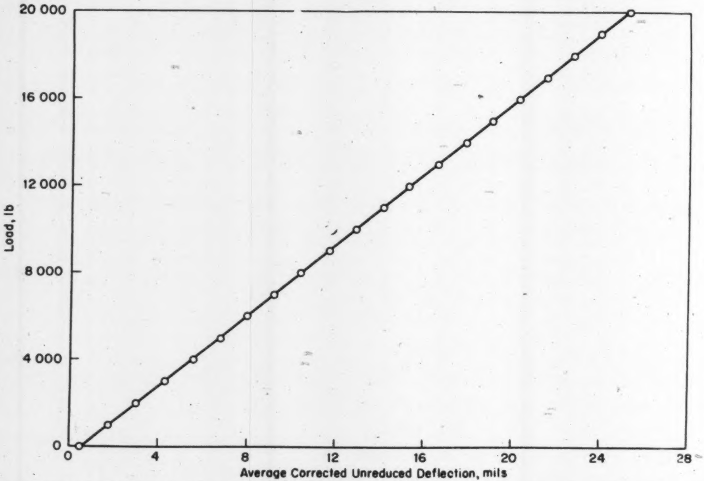


FIG. 5.—Typical Example of Load-Deflection Curve with Correction for Gage Calibration. First test with zero notch depth.

TABLE II.—SUMMARY OF DEFLECTIONS IN BENDING.

Test	Slot Depth, c, mils	N	Where: $P = 20{,}000$ lb $M = 46{,}250$ in.-lb				Where: $P = 10{,}000$ lb $M = 23{,}125$ in-lb			
			β_N, mils	$\beta_N - \beta_u$, mils	$\Delta\theta$	K	β_N, mils	$\beta_N - \beta_u$, mils	$\Delta\theta$	K
No. 1	0	0	24.70	$+0.01$	$+0.02 \times 10^{-4}$	0	12.45	-0.02	-0.04×10^{-4}	0
No. 2	0	0	24.68	-0.01	-0.02×10	0	12.49	$+0.02$	$+0.04 \times 10$	0
No. 3	0.200	0.100	25.24	0.55	1.19×10	2.6×10^{-9}	12.76	0.29	0.63×10	2.7×10^{-9}
No. 4	0.200	0.100	25.10	0.41	0.89×10	1.9×10	12.74	0.27	0.58×10	2.5×10
No. 5	0.400	0.200	26.65	1.96	4.24×10	9.2×10	13.44	0.97	2.10×10	9.1×10
No. 6	0.400	0.200	26.50	1.81	3.92×10	8.5×10	13.41	0.94	2.03×10	8.8×10
No. 7	0.610	0.305	23.51[a]	3.77	8.15×10	22.0×10	14.79	2.32	5.02×10	21.7×10
No. 8	0.610	0.305	23.65[a]	3.85	8.33×10	22.5×10	14.85	2.38	5.15×10	22.3×10
No. 9	0.800	0.400					17.02	4.55	9.84×10	42.5×10
No. 10	0.800	0.400					16.89	4.42	9.56×10	41.3×10
No. 11	1.000	0.500					20.68	8.21	17.76×10	76.8×10
No. 12	1.000	0.500					20.62	8.15	17.62×10	76.2×10
No. 13	1.000V[b]	0.500					20.80	8.33	18.02×10	77.8×10

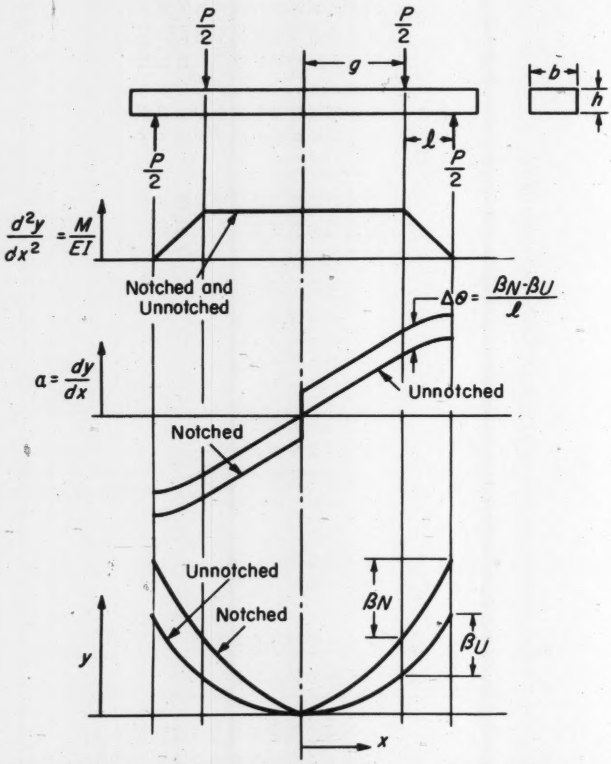
[a] For 16,000-lb load ($M = 37{,}000$ in.-lb).

[b] 45-deg V notch.

as shown in Fig. 6. K is defined as the $\Delta\theta$ per unit moment:

$$K = \frac{\Delta\theta}{M}$$

and has the physical meaning of excess compliance due to notching.



$\frac{P}{2}$
$\frac{P}{2}$
g
b
h
l
$\frac{P}{2}$
$\frac{P}{2}$
$\frac{d^2y}{dx^2} = \frac{M}{EI}$
Notched and Unnotched
$\Delta\theta = \frac{\beta_N - \beta_U}{l}$
$a = \frac{dy}{dx}$
Unnotched
Notched
Unnotched
Notched
β_N
β_U
y
x

FIG. 6.—Schematic Diagram Illustrating the Definitions of Bend Deflections and Angular Deflections.

The tension specimen, made from a heat-treated nickel-molybdenum-vanadium steel forging,[6] was $2\frac{1}{4}$ in. in diameter. It was provided with a circumferential 45-deg V notch[7] having a 3-mil root radius maximum. Deflection measurements were taken at nominal notch depths representing 0, 15, 30, 40, 50, and 60 per cent of cross-section removed. Minimum diameter was measured with a toolmaker's microscope to 0.001 in.

Deflection measurements were made with Huggenberger gages on opposite sides of the specimen and straddling the notch.

The Huggenberger gages had a nominal lever ratio of 2000, and a gage length of 4 in. was used. Since the actual lever ratio depends on clamping force (11), they were calibrated against curva-

---

[6] This forging was designated as "WD" in reference (8). The reference indicates the metallurgical properties of this steel.

[7] It will be shown for bending that a 45-deg V notch is equivalent to a narrow slot notch.

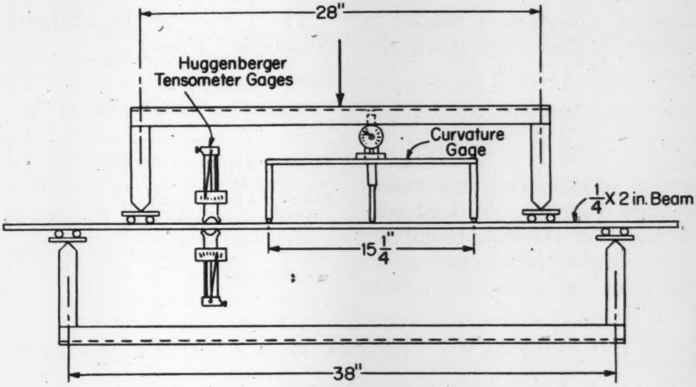

28"
Huggenberger Tensometer Gages
Curvature Gage
$\frac{1}{4}$ X 2 in. Beam
$15\frac{1}{4}$"
38"

FIG. 7.—Setup for Calibrating Huggenberger Gages.

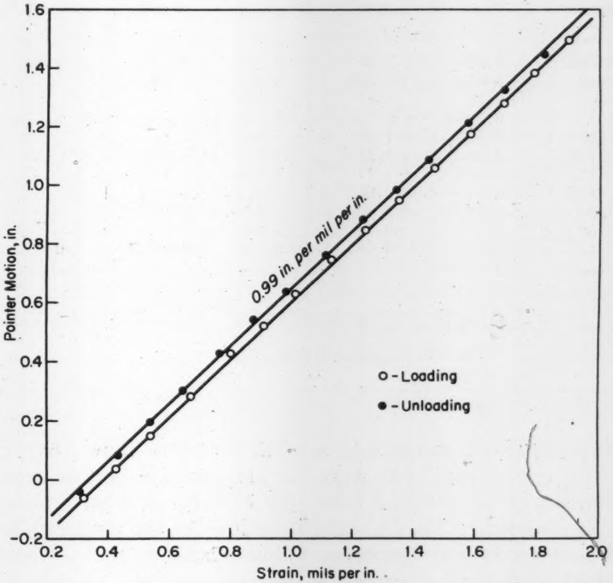


FIG. 8.—Calibration of Huggenberger Gage No. 635 on the Tension Side of the Beam.

ture of a beam, as shown in Fig. 7, using approximately the same clamping force as in the tests. Uniform curvature was insured by four point loading through frictionless loading points.

Strain e is related to radius of curvature r and beam thickness t by:

$$e = \frac{t}{2r}$$

and radius of curvature is related to chord length l and arc-to-chord distance δ by:

$$r = \frac{\delta^2 + l^2/4}{2\delta}$$

For purposes of stability, the frame of the curvature gage was provided with one ball support at one end at midbeam width and with two ball supports at the other end placed near the edges of the beam. The arc-to-chord distance was measured with a 0.001-in. dial gage, which furnished ample sensitivity for the long chord length used.

Figure 8 shows a typical calibration curve. The loading and unloading curves were parallel and offset from each other slightly because of the mechanical take-up in the lever system. The data points did not quite follow perfectly straight lines, but they were faired with straight lines, as shown in Fig. 8.

TABLE III.—CALCULATION OF REDUCED HUGGENBERGER DEFLECTIONS.

(2¼ in. Diameter Tension Bar, 49.8 per cent Notch)

Load, 1000 lb	Pointer Motion (Gage No. 636), in.	Pointer Motion (Gage No. 635), in.	Average Pointer Motion × 2, in.	An Arbitrary Linear Motion, in.	Average Reduced Motion × 2, in.	Faired Value of Reduced Motion, in.	Faired Value of Total Motion × 2, in.
0....	−0.085	−0.085	−0.17	−0.240	+0.07	−0.012	−0.252
2....	+0.04	+0.045	+0.085	+0.090	−0.005	....	....
4....	+0.22	+0.20	+0.42	+0.420	0	....	....
6....	+0.40	+0.365	+0.765	+0.750	+0.015	....	....
8....	+0.565	+0.515	+1.08	+1.080	0	....	....
10....	+0.745	+0.68	+1.425	+1.410	+0.015	....	....
12....	+0.925	+0.845	+1.77	+1.740	+0.03	....	....
14....	+1.085	+0.995	+2.08	+2.070	+0.01	....	....
16....	+1.26	+1.16	+2.42	+2.400	+0.02	....	....
18....	+1.425	+1.315	+2.74	+2.730	+0.01	....	....
20....	+1.60	+1.485	+3.085	+3.060	+0.025	+0.017	+3.077
Difference........							3.329

Average pointer motion: 1.6645 in. per 20,000 lb

Deflection (α): $\frac{1.6645}{2050} = 0.000812$ in. per 20,000 lb

The results of the calibrations are summarized below:

	Gage No. 635	Gage No. 636
Lever ratio according to manufacturer.........	2011	2084
Measured lever ratio:		
First measurement.......	1980	2140
Second measurement.....	2040	2040
Average.............	2010	2090

These results show that a particular calibration can deviate from the mean by as much as 2 per cent, probably depending on the clamping force, and so some scatter in the test results is to be expected. The average calibrations agreed with those furnished by the manufacturer.

As in the bend tests, reduced deflections for the notched tension tests were plotted against load, and the final value of deflection was obtained using values of reduced deflection read from the faired curves. In all cases, deflection was obtained by applying the average lever ratio to the average of the readings.

Table III and Fig. 9 show a typical calculation and a typical curve of reduced deflection *versus* load. The first point always had to be discarded because of the mechanical takeup in the gages. Slight curvature is again observed, as in the gage calibrations. However, this slight curvature does not seriously affect the calculation of energy release rates because, as discussed previously for bending, only the excess deflection due to notching affects the energy release rate. Table III shows the calculation of deflection for the data of Fig. 9.

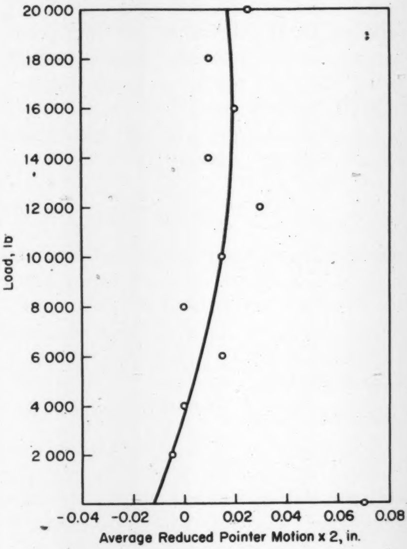


FIG. 9.—Reduced Huggenberger Deflections for 49.8 per cent Notch, $2\frac{1}{4}$ in. Diameter Tension Specimen. Deflections expressed as the sum of the pointer motions (average × 2) of the two gages. Four-inch gage length.

Tests to fracture were performed on notched tension bars of materials A and WC to determine critical energy release rate $\mathcal{G}_c$. All bars had threaded ends and 50 per cent sharp ($r < 0.003$ in.) 45-deg V notches. The WC bars were $2\frac{1}{4}$ in. unnotched diameter; and the A bars covered a range of unnotched diameters from $\frac{1}{4}$ to $2\frac{1}{4}$ in. Initial notch diameter was measured with a toolmaker's microscope.

The initial eccentricity was determined for each test by measuring elastic strains across the notch with Huggenberger gages. During these measurements and the subsequent test, the load was not allowed to fall to zero, so that the eccentricity would not change accidentally. Strains on opposite sides were measured, first in the side-to-side plane through the axis and then in the front-to-back plane. Fairly good concentricity was obtained by manual shifting of the fixture string with respect to the ball joints, but complete concentricity could not be attained because of slight imperfections in the threaded joints. In most of the tests, the maximum elastic surface strain was about 10 per cent higher than the centerline strain (see Table VI). The ratio of maximum surface strain e_{sm} to centerline strain e_{cl} is given in terms of the measured strains by

$$e_{sm}/e_{cl} = \sqrt{\frac{e_{a\ \max}}{e_{a\ \min}} + \frac{e_{b\ \max}}{e_{b\ \min}}}$$

where a and b are two perpendicular planes through the axis.

During the test, the change in distance from the notch root on one side to the cylindrical surface on the other side was measured continuously by means of a micrometer provided with a sharp 40-deg chisel anvil. This chisel anvil fitted into the notch on one side and the flat-ended spindle straddled the notch on the other side. The change in notch diameter could be determined by doubling this measured change, since no change in unnotched diameter occurred.

From the load and the change in notch diameter, curves of average axial stress (S_a) *versus* average transverse strain (e_t),

$$S_a = \frac{4P}{\pi d^2}$$

$$e_t = \frac{\Delta d}{d}$$

can be plotted, where d is notch diameter and P is load. The offset from the elastic portion of such stress-strain curves may be considered as "plastic strain," and the plastic strain at fracture, obtained by a short extrapolation, was taken as the "notch ductility." Actually this notch ductility is an average value that bears little resemblance[8] to the level of local strain at fracture, but it does serve as a measure of how extensively plastic flow has occurred.

ENERGY RELEASE RATE IN NOTCH BENDING

Figure 4 shows that the corrected load-deflection curve for the unnotched beam is not quite straight. The hook on this curve is probably associated in some way with the clamping of the spring steel connectors between the specimen and the measuring bars. In other investigations (6), such an arrangement has been found to affect high-precision deflection measurements. If the corrected data of Fig. 4 is faired with a straight line (dashed), we find:

$$\Delta y = (0.23 - 0.58)(10^{-3}) + (20{,}000)(1.25 \times 10^{-6}) = 24.65 \times 10^{-3}$$

Substituting this deflection in the equation for beam bending (Appendix II):

$$\beta_u = \frac{Pl}{2bh^3E}\,[4l(3g + l) + 3h^2(1 + \mu)]$$

gives a modulus value of 31,300,000 psi, which is close to the value 29,700,000 psi measured in tension by means of the Huggenberger gages. In the above equation, β_u is the deflection under the loads relative to the supports for four-point loading, μ is Poisson's ratio (assumed to be 0.3), and the other quantities are defined by Fig. 6.

As shown in Appendix III, the energy release rate for a beam is:

$$\mathcal{G} = \frac{aS_n^2}{E}\,F(N) \qquad (1)$$

where a is net beam depth and N is the ratio of slot depth c to gross beam depth h. The function $F(N)$ has to be determined experimentally from the extra compliance (K) due to notching (Table II):

$$F(N) = \frac{Eba^3}{36}\left(\frac{dK}{dc}\right)$$

$F(N)$ can be determined by plotting K from Table II *versus* c and measuring the slope. The immediate objective is to compare with Bueckner's analytical expression (5):

$$\mathcal{G} = \frac{S_n^2a(1 - \mu^2)}{E}\,f(N)$$

and since slope measurements are not very accurate, this comparison will be made in a different way. If the analytical $\mathcal{G}_A$ and experimental $\mathcal{G}_E$ are to be equal:

$$\mathcal{G}_E = \mathcal{G}_A$$

$$\frac{aS_n^2}{E}\,[F(N)] = \frac{S_n^2a(1 - \mu^2)}{E}\,f(N)$$

$$\frac{ba^4S_n^2}{36}\left(\frac{dK}{dc}\right) = \frac{S_n^2a(1 - \mu^2)}{E}\,f(N)$$

$$\frac{ba^3}{36}\left(\frac{dK}{dc}\right) = \frac{(1 - \mu^2)}{E}\,f(N)$$

$$dK = \frac{36(1 - \mu^2)f(N)dc}{Eba^3}$$

---

[8] At the first deviation from linearity of such stress-strain curves there has been plastic strain in considerable *degree,* but of exceedingly limited *extent* (9).

Noting that only (dK/dc), $f(N)$, and a are functions of c, and integrating:

$$K = \int_0^K dK = \frac{36(1 - \mu^2)}{Eb} \int_0^c \frac{f(N)dc}{a^3}$$

Thus, the area under the curve of $[f(N)/a^3]$ plotted against c multiplied by $[36(1 - \mu^2)/Eb]$ should equal the experimental values of K if the experimental and analytical expressions agree. The comparison in Fig. 10[9] shows that the two expressions do agree very closely up to a notch depth of 0.400 in., and moderately well up to 1 in. Irwin (10) shows a comparison which also indicates close agreement, but the experimentation from which the result is derived is not discussed, and the experimental result is for three-point rather than four-point loading.

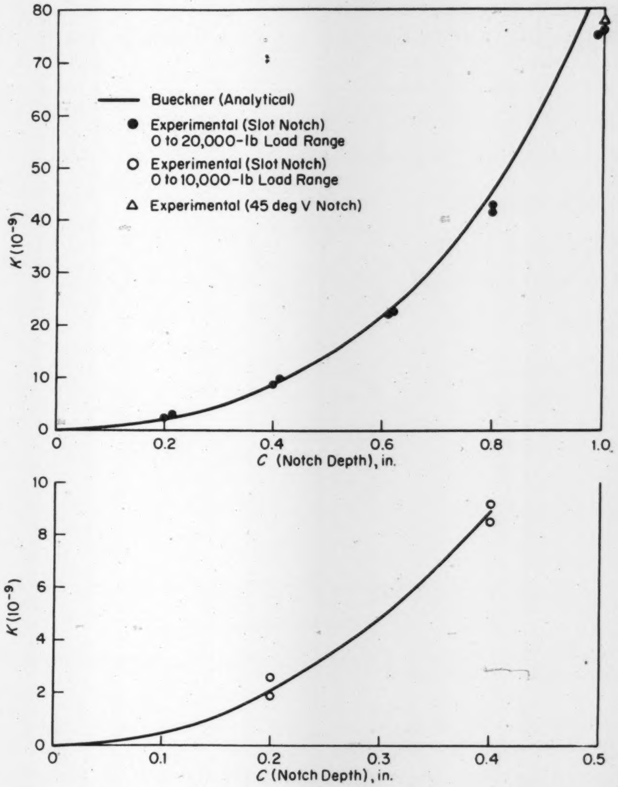


FIG. 10.—Excess Deflection (Due to Notching) per Unit Moment, K, as a Function of Notch Depth.

Figure 11 shows the values of $F(N)$ in Eq 1 as derived from the slopes of the experimental K *versus* c curve in Fig. 10 and the experimental values $b = 2$ in. and $a = 2$ in. $- c$. This curve is to be used in connection with Eq 1 in determining energy release rate for notched beams. For comparison, the analytical solutions referred to by Winne and Wundt are also shown. By means of this comparison, the same conclusion is reached as that drawn from Fig. 10: The analytical results agree with the experimental results up to about 20 per cent notch depth[10] and deviate somewhat for deeper notches. The experimental results indicate that $F(N)$ does not increase beyond about 40 per cent notch depth. Winne and Wundt also showed the $F(N)$ curve horizontal, but not until beyond 50 per cent notch depth.

---

[9] In Fig. 10, the small effects at small notch depths have been defined somewhat more accurately by using a longer load-deflection curve. Larger loads could be used for these shallow notches because the strength is higher for shallow notches. Table II shows the different K values based on both long and short load ranges.

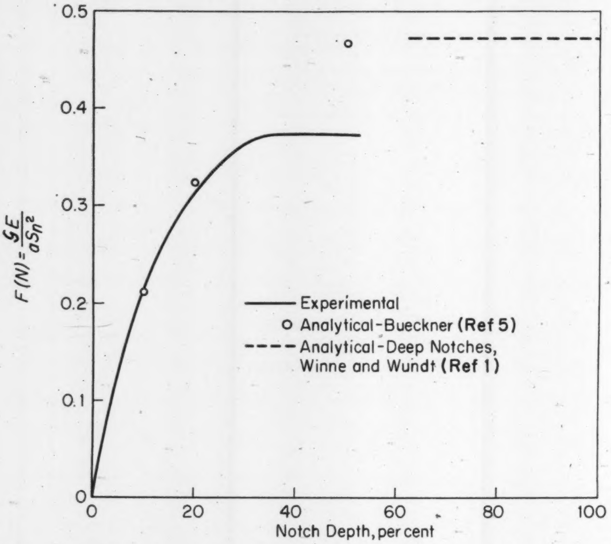


FIG. 11.—Values of $F(N)$ (that is a Function of Notch Depth N) to be Used in Eq 1 to Obtain Energy Release Rate in Notch Bending.

To see whether analytical solutions for slot notches (cracks) can be applied to specimens containing the more easily machined V notches that are commonly employed in laboratory testing, a load-deflection curve was measured for one bend specimen containing a sharp 50 per cent 45-deg V notch. The excess compliance K for this test is shown by the triangle in Fig. 10. This result is so close to the results for the narrow (16-mil) slot notches (filled circles) that we may conclude that sharp V notches with included angles less than 45 deg are equivalent to narrow slot notches. This result might be expected if one recognizes that the metal adjacent to the top of the slot is essentially unstressed, and so it matters little whether or not it is there. Considering this result, the tension bars for load-deflection measurements were provided with V notches instead of slot notches.

---

[10] In Fig. 11, the agreement at 20 per cent notch does not appear to be quite as good as is indicated in Fig. 10. This discrepancy is due to the fact that the construction of Fig. 11 requires graphical differentiation to obtain dK/dc, and therefore is less accurate than the comparison in integral form in Fig. 10.

APPLICATION TO FRACTURE IN NOTCH BENDING

As an example of the use of Eq 1 to determine the material property $\mathcal{G}_c$ from test data, consider the following result of a notch bend test on material A. This specimen was 2 in. square and was provided with a sharp 50 per cent 45-deg V notch. It was tested with the same support and loading spans as in determining load-deflection curves. For the breaking load of 14,400 lb, the nominal stress is:

$$S_n = \frac{M}{I/c}$$

$$M = \frac{Pl}{2} = \frac{14{,}400 \times 4.625}{2} = 33{,}300 \text{ in-lb}$$

$$\frac{I}{c} = \frac{bh^2}{6} = \frac{2 \times 1^2}{6} = \frac{1}{3}$$

$$S_n = 3 \times 33{,}300 = 100{,}000 \text{ psi}$$

Substituting in Eq 1 and reading a value of 0.373 from Fig. 11 for $N = 50$ per cent, a value of $\mathcal{G}_c$ of:

$$\mathcal{G}_c = \frac{1 \times 100{,}000^2 \times 0.373}{29{,}700{,}000}$$

$$= 126 \text{ in-lb per sq in.}$$

is obtained. This is very close to the value that can be obtained by applying Eq 1 to earlier notch bend tests (9) on the same material, but using 20 per cent notches. Figure 12 shows that the Griffith-Irwin inverse square root law applies for values of gross beam depth of $\frac{3}{4}$ in. or over, and Table IV shows that these specimens yield an average value of $\mathcal{G}_c$ of 131. This value is very close to the value of 126 for the 2-in. square bend specimen with 50 per cent notch.

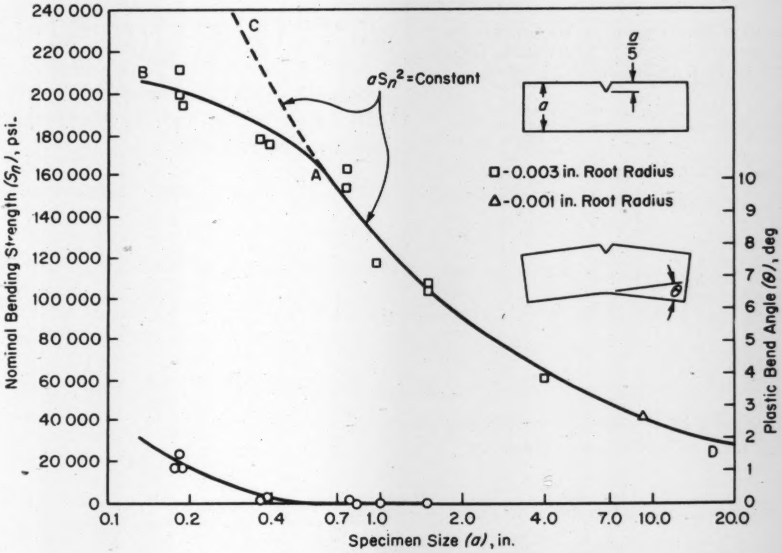


FIG. 12—Correlation of Notch Bend Data with the Size Effect Predicted by the Griffith-Irwin Theory.

ENERGY RELEASE RATE IN NOTCH TENSION

Table V summarizes the deflection measurements for various notch depths and shows the calculation of unit excess compliance due to notching:

$$K = \frac{\delta_{\text{notched}} - \delta_{\text{unnotched}}}{P}$$

as required for determining energy release rate.

TABLE IV.—ENERGY RELEASE RATE FOR LARGE SQUARE NOTCHED BEAMS WITH 20 PER CENT NOTCH.

Gross Beam Depth, in.	Notch Radius, mils	Nominal Bending Strength, kips	$\mathcal{G}_c$ in-lb per sq in.
¾	3	162	152
¾	3	153	135
1	3	116.5	105
1½	3	107	133
1½	3	102	122
2	5	103	165
4	3	60.3	113
8.6	1	43	122
Average			131

Appendix IV shows that energy release rate $\mathcal{G}$ for notch tension is given by:

$$\mathcal{G} = \frac{S_n^2 d}{E} F(N) \qquad \text{(2)}$$

where d is notch diameter and:

$$F(N) = -\frac{\pi^2 d^3 E}{32}\left(\frac{dK}{dA}\right) \qquad \text{(3)}$$

Figure 13[11] shows K plotted against A for purposes of graphical differentiation.

Figure 14 shows $F(N)$ as determined by graphical differentiation and Eq 3. In this figure, the data is presented in a form suitable for using in conjunction with Eq 2 to obtain $\mathcal{G}_c$ from notch tension tests. Figures 13 and 14 also show, for comparison, the analytical solutions of Irwin and Bueckner. Irwin (2) proposed the relationship:

$$\mathcal{G} = \frac{\pi\sigma^2 a}{E}\left[1 + \left(\frac{a}{R_0 - a}\right)\left(1 - \frac{2}{\pi}\right)\right] \qquad \text{(4)}$$

where σ is stress in the unnotched section, a is notch depth, and R_0 is unnotched radius. Noting that:

$$P = SA = \frac{\pi D^2 \sigma}{4} = \frac{\pi d^2 S_n}{4}$$

$$\sigma = \frac{S_n d^2}{D^2}$$

$$2(R_0 - a) = d$$

$$a = \frac{D}{2}\left(1 - \frac{d}{D}\right)$$

$$N = 1 - \left(\frac{d}{D}\right)^2$$

Equation 4 becomes:

$$\left.\begin{aligned} \mathcal{G} &= \frac{S_n^2 d}{E}\,[f_1(N)]; \\ f_1(N) &= (1 - N)\,(1 - \sqrt{1 - N}) \\ &\quad (\pi/2 - 1 + \sqrt{1 - N}) \end{aligned}\right\} \qquad \text{(5)}$$

Bueckner (5) has proposed the relationship:

$$\mathcal{G} = \frac{\pi(1 - \mu^2) D \sigma^2 m^2}{4E}\left(\frac{D}{d}\right)^4 \qquad \text{(6)}$$

where σ is defined as in Eq 4 and m was determined numerically as:

---

[11] The scattering in Fig. 13 is considerably larger than that for bending as shown in Fig. 10, because of the shorter travel of the extensometer during the test as compared to the sensitivity, and the effect of this shorter travel on the accuracy of the relatively small differences between unnotched and notched extensions (see Table V). For the dial indicator type of deflection gage used in the bend tests, this travel was about ten times longer than that for the Huggenberger gages used in the tension tests. Thus greater accuracy could be obtained by developing a dial indicator type of extensometer. Also some additional accuracy might be obtainable if a shorter gage length were used, while still completely enclosing the region of disturbed stress distribution in the neighborhood of the notch.

$\frac{d}{D} = \sqrt{1 - N}$	m
0.1	0.21
0.2	0.30
0.3	0.37
0.4	0.42
0.5	0.46
0.6	0.47
0.7	0.46
0.8	0.435
0.9	0.39

;

Equation 6 can be rewritten as:

$$\mathcal{G} = \frac{S_n^2 d}{E} [f_2(N)];$$

$$f_2(N) = \frac{\pi(1 - \mu^2)m^2}{4\sqrt{(1 - N)}} \quad \ldots\ldots\ldots (7)$$

The quantities f_1 and f_2 in Eqs 5 and 7, that is, $\mathcal{G}E/S_n^2d$, have been plotted in Fig. 14 as the dot-dash line and dashed line respectively. The empty circles in Fig. 14 correspond to the values of m tabulated above.

TABLE V.—SUMMARY OF NOTCH TENSILE EXTENSIONS ON 4-IN. GAGE LENGTH.

Notch Diameter, in.	Notch Area, sq in.	Notch Depth, per cent	Load, 1000 lb	Extension, mils	Average Extension, mils	Extension Change Due to Notch, mils	K (Extension Change per Unit Load), in/lb
2.250	3.980	0	16	0.548	0.546	...	0
2.250	3.980	0	16	0.543		...	0
2.250	3.980	0	18	0.618	0.612	...	0
2.250	3.980	0	18	0.607		...	0
2.250	3.980	0	20	0.689	0.680	...	0
2.250	3.980	0	20	0.671		...	0
2.091	3.437	13.6	20	0.690	...	0.010	0.5×10^{-9}
2.091	3.437	13.6	20	0.688	...	0.008	0.4×10
1.902	2.843	28.5	18	0.673	...	0.061	3.4×10
1.902	2.843	28.5	18	0.666	...	0.054	3.0×10
1.763	2.442	38.6	16	0.590	...	0.044	2.75×10
1.763	2.442	38.6	16	0.602	...	0.056	3.5×10
1.763	2.442	38.6	20	0.738	...	0.058	2.9×10
1.595	1.999	49.8	20	0.819	...	0.139	6.95×10
1.595	1.999	49.8	20	0.812	...	0.132	6.6×10
1.427	1.600	59.8	18	0.779	...	0.167	9.3×10
1.427	1.600	59.8	18	0.771	...	0.159	8.8×10

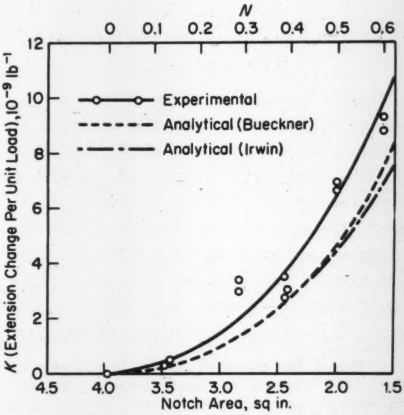


FIG. 13.—Experimental and Analytical Values of K as a Function of Notch Area.

The comparison between analytical and experimental results in Fig. 14 is not quite satisfactory, because the coefficient of S_n^2d/E requires graphical differentiation of the faired K *versus* A curve of Fig. 13, and consequently the analytical solutions cannot be compared with the actual measured values. Consequently, K values were determined for the analytical solutions by integration and plotted in Fig. 13 for comparison with the measured values.

The variation of K with notch area for the two analytical solutions can be obtained by integration, as follows.

Comparison of Eqs 2, 3, 5, and 7 shows that dK/dA must be related to f_1 and f_2 by:

$$\frac{dK}{dA} = -\frac{32f_1(N)}{\pi^2 d^3 E}$$

$$\frac{dK}{dA} = -\frac{32f_2(N)}{\pi^2 d^3 E}$$

Noting that only d and f are functions of A and integrating:

$$K = -\frac{32}{\pi^2 E}\int_{A_0}^{A} \frac{dA}{d^3} f_1(N)$$

$$K = -\frac{32}{\pi^2 E}\int_{A_0}^{A} \frac{dA}{d^3} f_2(N)$$

These values of K have been plotted in Fig. 13 for the experimental values of E (modulus) and A (unnotched area):

$$E = 29{,}700{,}000 \text{ psi}$$
$$A_0 = 3.98 \text{ sq in.}$$

The resulting curves, shown as dot-dash for Irwin's solution and dashed for Bueckner's solution, lie below the lowest experimental points. Therefore, Fig. 13 bears out the conclusion, already indicated in Fig. 14, that the two analytical results agree up to about 40 per cent notch depth, but neither of the analytical results agrees with the experimental results.

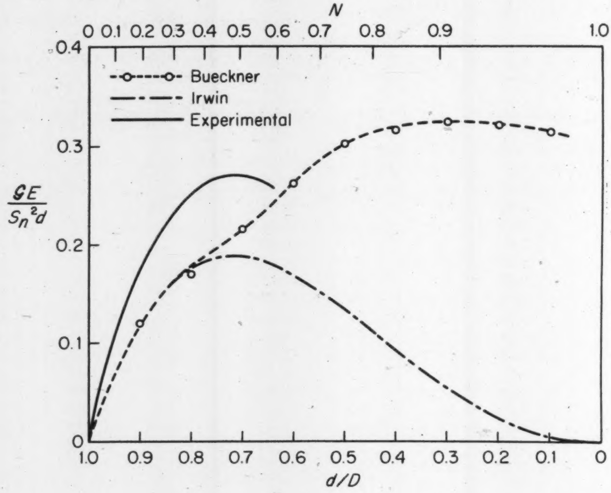


FIG. 14.—Coefficient of $S_n^2 d/E$ in the Expression for Energy Release Rate $\mathcal{G}$.

FRACTURE BEHAVIOR IN NOTCHED TENSION TESTS

Figure 15 shows examples of stress-strain curves to fracture for notch tension tests on material A. In these curves, stress is average longitudinal stress over the minimum cross-section and strain is average transverse strain along a notch diameter. For each test an elastic line has been drawn whose slope represents the average for all the tests. The elastic behavior[12] of notched

---

[12] **Based on more sensitive measurements, Sachs (12) reports an elastic slope of the longitudinal stress – transverse strain curve that is seven times larger than the slope shown in Fig. 15.**

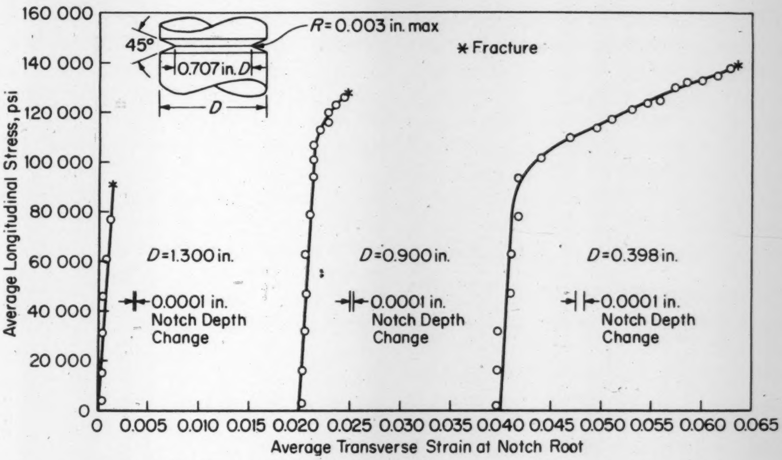


FIG. 15.—Typical Examples of Stress-Strain Curves for Notched Tension Test Specimens.

TABLE VI.—SUMMARY OF NOTCH TENSION TEST RESULTS.

Material	Un-notched Diameter, in.	Notch Diameter, in.	Net Area, sq in.	Notch Area, per cent	P_{max}, lb	S_n, psi	e_{sm}/e_{cl}	Plastic Notch Depth Change at Fracture, in.	Plastic Notch Strain, per cent	$\mathcal{G}_c$ in-lb per sq in.
A..........	0.250	0.1775	0.02475	50	3 750	151 000	1.16	0.0025	2.8	...
A..........	0.252	0.1781	0.02492	50	3 720	149 000	1.16	0.0033	3.7	...
A..........	0.402	0.2838	0.0633	50	9 000	142 000	1.10	0.00215	1.5	...
A..........	0.3985	0.2852	0.0639	49	8 880	139 000	1.05	0.0031	2.2	...
A..........	0.599	0.4193	0.1380	51	18 100	131 000	1.09	0.0015	0.7	...
A..........	0.598	0.4203	0.1386	51	18 800	136 000	1.07	0.0018	0.9	...
A..........	0.900	0.6360	0.3177	50	40 400	127 000	1.10	0.00095	0.3	94
A..........	0.899	0.6339	0.3155	50	39 000	124 000	1.08	0.0009	0.3	89
A..........	1.3005	0.9200	0.6647	50	71 400	108 000	1.08	0	0.0	98
A..........	1.300	0.911	0.6520	51	59 800	92 000	1.06	0	0.0	71
A..........	2.250	1.593	1.993	50	146 000	73 000	1.00	0.0002	0.0	78
A..........	2.250	1.600	2.011	49	148 000	74 000	1.11	...	...	80
WC........	2.250	1.591	1.989	50	143 000	72 000	1.02	0.0005	0.1	76
WC........	2.250	1.590[a]	1.986	50	190 000	96 000	1.07	0.0005	0.1	143

[a] 4-mil root radius.

specimens should not be inferred from this slope value, however, because of the very low sensitivity of the measurements (smallest perceptible change of notch depth of 0.0001 in. is indicated in the figure).

Figure 15 shows that as the size increases the failure occurs earlier, that is, with a lower average stress and a considerably lower "ductility" or average transverse plastic strain. (Size can only affect the location of the failure point on the curve; the Law of Similitude (9) requires that the shape of the stress-strain curve be independent of size.) The effects of specimen size are summarized

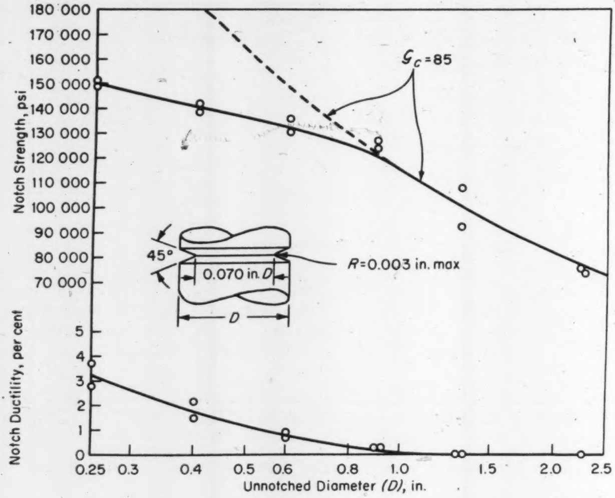


FIG. 16.—Notch Tension Test Results on Material A. Forty-five degree, 50 per cent sharp V-notches.

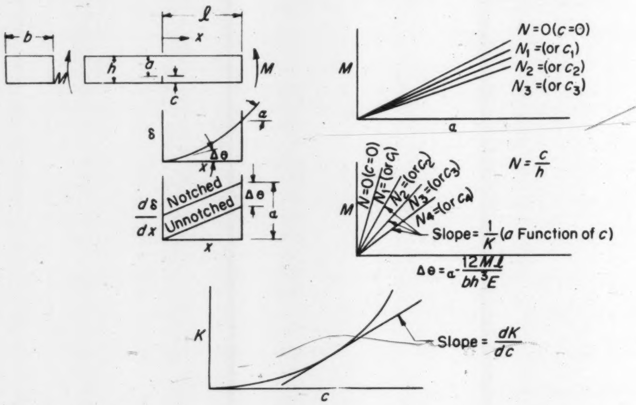

b
l
x
M
h
a
c
M
δ
a
Δθ
x
dδ/dx
Notched
Unnotched
Δθ
a
x
M
N=0(c=0)
N₁=(or c₁)
N₂=(or c₂)
N₃=(or c₃)
a
M
N=0(c=0)
N₁=(or c₁)
N₂=(or c₂)
N₃=(or c₃)
N₄=(or c₄)
N = c/h
Slope= 1/K (a Function of c)
Δθ = a - 12Ml/bh³E
K
Slope = dK/dc
c

FIG. 17.—Schematic Diagram Showing Conditions for Experimental Determination of Energy Release Rate $\mathcal{G}$.

in Table VI and Fig. 16. The comparatively low scattering of results under these notch brittle conditions is a reflection of the low initial eccentricity as indicated by the ratio e_{sm}/e_{cl} in Table VI.

At the larger sizes, the notch strength values conform to the inverse square root relationship indicated by Eq 2 (or at least do not disagree with this relationship); at the smaller sizes, the notch strength values lie below the theoretical line because of extensive plastic flow at the notch. The extent of this plastic flow is indicated by the notch ductility plotted in the lower part of the figure. Note that the strength conforms to Eq 2 when the notch ductility is negligibly small (less than the scatter), and deviates progressively from Eq 2 as the notch ductility increases. The same general behavior was observed for bending (Fig. 12).

In Fig. 16, the inverse square root line fairing the points at large size corresponds to a $\mathcal{G}_c$ value of 85. This value was obtained by applying Eq 2 to the data for the three largest sizes using $F(N) = 0.27$ from Fig. 14 for 50 per cent notch, and averaging the resulting values of $\mathcal{G}_c$ (see Table VI). The value 85 is considerably smaller than the value of 131 for notch bending (see Fig. 12 and Table IV). If the notch tensile strength had been predicted from the bend test results, the values would have been in error by:

$$\sqrt{\frac{131}{85}} = 1.24$$

or 24 per cent. This discrepancy is somewhat greater than the 17 per cent correlation error between notch bending ($\mathcal{G}_c = 252$) and notch disk bursting ($\mathcal{G}_c = 184$) (13) for material WC. Two notch tension tests on material WC (Table VI) provide additional correlations among different types of tests as shown below:

Material	Type of Test	Average $\mathcal{G}_c$	Stress Error, per cent
A..........	Bending	131	24
A..........	Tension	85	
WC.........	Bending	252	17 51
WC.........	Disk bursting	184	29
WC.........	Tension	110	

These additional correlations do not put the usefulness of the Griffith-Irwin concept in as favorable a light as previous correlations. In other words, the test results of Table VI would suggest that correlation of different types of tests by means of the Griffith-Irwin concept should be carried out with somewhat greater caution than earlier results would indicate.

CONCLUSIONS

1. It is feasible to determine energy release rate $\mathcal{G}$ experimentally for notch bending and notch tension, if certain precautions are observed.

2. In notch bending, the experimental and analytical values of $\mathcal{G}$ agree up to 20 per cent notch depth, and diverge for deeper notches.

3. In notch tension, the experimental values of $\mathcal{G}$ are about 40 per cent higher than the analytical values over most of the range of notch depth that was investigated.

4. The Griffith-Irwin concept of a critical energy release rate accurately predicts the effects of notch depth and specimen size if extensive plastic flow has not occurred.

5. Strength discrepancies of 25 to 50 per cent arise if the Griffith-Irwin concept is used to correlate notch tension tests with other types of tests.

Acknowledgment:

The author is indebted to R. Sakamoto and P. H. Grissom, of the General Electric Co., Medium Steam Turbine, Generator, and Gear Department, for their interest and sponsorship of this investigation; and to P. R. Paslay and S. Yukawa for their contributions to the development of physical concepts and mathematical analyses. The author would also like to thank S. N. Shafer and E. P. Kexel for their assistance with the experimentation.

REFERENCES

(1) D. H. Winne and B. M. Wundt, "Application of the Griffith-Irwin Theory of Crack Propagation to the Bursting Behavior of Disks, Including Analytical and Experimental Studies," *Preprint No. 57–A–249*, Am. Soc. Mechanical Engrs. (1957).

(2) G. R. Irwin, "Onset of Fast Crack Propagation in High-Strength Steel and Aluminum Alloys," Naval Research Laboratory Report No. 4763, May 24, 1956.

(3) A. A. Griffith, "The Phenomena of Rupture and Flow in Solids," *Philosophical Transactions*, Royal Soc. (London), Series *A*, Vol. 221, pp. 163–198 (1920).

(4) G. R. Irwin and J. A. Kies, "Critical Energy Rate Analysis of Fracture Strength," *Welding Journal*, Vol. 33, p. 193-S (1954).

(5) H. F. Bueckner, "The Stress Concentration of a Notched Bar in Bending," *Data Folder 51TG153*, General Electric Co., June 28, 1957.

(6) J. D. Lubahn, "Constant Volume Condition at Small Plastic Strains," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 1327 (1958).

(7) C. Schabtach, A. W. Rankin, E. L. Fogleman, and D. H. Winne, "Report of the Investigation of Two Generator Rotor Fractures," *Preprint No. 55–A–208* Am. Soc. Mechanical Engrs. (1957).

(8) D. R. Deforest, D. L. Newhouse, and B. R. Seguin, "Progress in the Development of Steam Turbine-Generator Materials," *Preprint No. 57–A–208* Am. Soc. Mechanical Engrs. (1959).

(9) J. D. Lubahn and S. Yukawa, "Size Effects in Slow Notch Bend Tests of a Ni-Mo-V Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 661 (1958).

(10) G. R. Irwin, J. A. Kies and H. L. Smith, "Fracture Strengths Relative to Onset and Arrest of Crack Propagation," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 640 (1958).

(11) R. W. Vose, "Characteristics of the Huggenberger Tensiometer," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, p. 862 (1934).

(12) G. Sachs, J. D. Lubahn, and L. J. Ebert, "Notched Bar Tensile Test Characteristics of Heat Treated Low Alloy Steels," *Transactions*, Am. Soc. for Metals, Vol. 33 p. 340 (1944).

(13) J. D. Lubahn, "Correlation of Tests Using the Congruency Principle," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 678 (1958).

APPENDIX I

ESTIMATED BEAM STRENGTH FOR VARIOUS NOTCH DEPTHS

From reference (1), the nominal notch bending strength S_{nf} is related to the critical energy release rate $\mathcal{G}_c$ by:

$$\mathcal{G}_c = \frac{S^2_{nf}a(1 - \mu^2)}{E} f\left(\frac{c}{h}\right)$$

where c is notch depth, h is gross beam depth, a is net beam depth, and f is given in Fig. 16 of reference (1). For $c/h = 0.2, f = 0.358$. Applying to specimens with sharp notches and large size and 20 per cent notch depth, and made of the material A (see Fig. 8 of reference 9), an average $\mathcal{G}_c$ of 131 in-lb per sq in. is obtained (Table IV). This value of $\mathcal{G}_c$ may now be used in the same formula to obtain the load at fracture for various notch depths:

Slot depth, c, in.	c/h	$f(c/h)$	a, in.	S_{nf}, psi	$M = \frac{SI}{c}$, in-lb	$P = \frac{2M}{1}$, lb
0.2.........	0.1	0.233	1.8	101 000	109 000	47 000
0.4.........	0.2	0.358	1.6	87 000	74 000	32 000
0.6.........	0.3	0.434	1.4	84 000	55 000	24 000
0.8.........	0.4	0.486	1.2	86 000	40 000	18 000
1.0.........	0.5	0.513	1.0	91 000	31 000	13 000

$\frac{I}{c} = \frac{ba^2}{6}$, and the other quantities are defined in Fig. 6.

APPENDIX II

UNNOTCHED BEAM DEFLECTIONS FOR FOUR POINT LOADING

From $x = 0$ to $x = g$ (Fig. 6), the moment

$$M = \frac{Pl}{2}$$

is constant and the shearing force is zero. Then:

$$\frac{dy}{dx} = \int_0^x \frac{Pl}{2EI} = \frac{Plx}{2EI}$$

$$y = \int_0^x \frac{Plx}{2EI} = \frac{Plx^2}{4EI}$$

where slopes and deflections are measured relative to conditions at midspan and $I = bh^3/12$. In particular, at $x = g$,

$$\left(\frac{dy}{dx}\right)_g = \frac{Plg}{2EI} \quad \text{and} \quad y_g = \frac{Plg^2}{4EI} = \frac{6Plg^2}{2bh^3E}$$

From $x = g$ to $x = (g + l)$, the shearing force V is constant at $P/2$ and the moment M is:

$$M = \frac{P}{2}(l + g - x)$$

The slope of the deflection curve is:

$$\frac{dy}{dx} = \left(\frac{dy}{dx}\right)_g + \int_g^x \frac{Mdx}{EI} + \frac{2KV(1 + \mu)}{AE}$$

where $K = 3/2$, $A = bh$, and μ is Poisson's ratio. Substituting:

$$\frac{dy}{dx} = \frac{Plg}{2EI} + \frac{P(l + g)}{2EI}\int_g^x dx - \frac{P}{2EI}\int_g^x x\,dx + \frac{3P(1 + \mu)}{2bhE}$$

$$\frac{dy}{dx} = \frac{P}{4EI}\left[2lx - (g - x)^2\right] + \frac{3P(1 + \mu)}{2bhE}$$

Also from $x = g$ to $x = (g + l)$:

$$y = y_g + \int_g^x \frac{dy}{dx}$$

$$y = \frac{Plg^2}{4EI} + \left[\frac{3P(1 + \mu)}{2bhE} - \frac{Pg^2}{4EI}\right]\int_g^x dx + \frac{P(l + g)}{2EI}\int_g^x x\,dx = \frac{P}{4EI}\int_g^x x^2\,dx$$

$$y = \frac{P}{12EI}\left[3lx^2 + (g - x)^3\right] + \frac{3P(1 + \mu)}{2bhE}\left[x - g\right]$$

In particular, at $x = (g + l)$:

$$y = \frac{Pl}{2bh^3E}\left[2(3g^2 + 6gl + 2l^2) + 3h^2(1 + \mu)\right]$$

The deflection under the load relative to the supports is the difference in y values at $x = g$ and $x = (g + l)$:

$$\Delta y = y_{g+l} - y_g = \frac{Pl}{2bh^3E}\left[4l(3g + l) + 3h^2(1 + \mu)\right]$$

APPENDIX III

DERIVATION OF ENERGY RELEASE RATE FOR NOTCH BENDING IN TERMS OF LOAD-DEFLECTION CURVES FOR NOTCHED SPECIMENS

In determining energy release rate experimentally for a notched beam, we start with an unnotched beam of depth h and measure the load-deflection curve. Then a shallow slot is machined into the beam and the load-deflection curve is measured again. Finally, a series of load-deflection curves is obtained for progressively deeper slots. Specifically, these curves would consist of bending moment plotted against the angular deflection α at one end of the beam. A different curve would be obtained for each slot depth c, that is, for each value of the nondimensional "notch depth" $N = \frac{c}{h}$ as shown in the upper right of Fig. 17.

Actually, in the case of the beam it is not the total angular deflection α that we are interested in, but rather that portion $\Delta\theta$ that is due to the notch. As shown in Fig. 17, α depends on the length of the beam, but $\Delta\theta$ does not. The quantities $\Delta\theta$ and α can be related by means of simple beam theory:

$$\frac{dy}{dx} = \int\left(\frac{d^2y}{dx^2}\right)dx = \int\frac{Mdx}{EI}$$

$$\alpha = \Delta\theta + \int_0^l \frac{12Mdx}{bh^3E} = \Delta\theta + \frac{12Ml}{bh^3E} \quad \text{.......................(8)}$$

The elastically-stored energy at a particular slot depth c and angular deflection α is $M\alpha$, and that at a slightly greater slot depth $(c + dc)$ and the same angular deflection α is

$$[M + (\partial M/\partial c)_\alpha][\alpha]$$

The derivative $(\partial M/\partial c)_\alpha$ has been computed at constant α so that no work will be done on the specimen while increasing c, and the energy released by deepening the slot will be *stored energy*, as required by the definition of energy release rate $\mathcal{G}$. While the slot depth is increasing from c to $c + dc$, the slot area increases from bc to $b(c + dc)$. Now we can write the energy release rate (with respect to slot area) as:

$$\mathcal{G} = \frac{M\alpha - [M + (\partial M/\partial c)_\alpha\, dc][\alpha]}{b(c + dc) - bc} = -\frac{\alpha(\partial M/\partial c)_\alpha}{b}$$

This can be expressed in terms of the slope $1/K$ of the M *versus* $\Delta\theta$ curves by substituting Eq 8 and performing the indicated differentiation:

$$\frac{1}{K} = \frac{M}{\Delta\theta}$$

$$\Delta\theta = KM$$

$$\alpha = KM + \frac{12Ml}{bh^3E} = M\left(K + \frac{12l}{bh^3E}\right)$$

$$M = \frac{\alpha}{K + \dfrac{12l}{bh^3E}}$$

$$\left(\frac{\partial M}{\partial c}\right)_\alpha = -\frac{\alpha(\partial K/\partial c)_\alpha}{\left(K + \dfrac{12l}{bh^3E}\right)^2}$$

$$\mathcal{G} = \frac{\alpha^2(\partial K/\partial c)_\alpha}{b\left(K + \dfrac{12l}{bh^3E}\right)^2} = \frac{M^2}{b}\left(\frac{\partial K}{\partial c}\right)_\alpha$$

or, in terms of nominal stress $S_n = Mc/I = 6M/ba^2$:

$$\mathcal{G} = \frac{ba^4S_n^2}{36}\left(\frac{\partial K}{\partial c}\right)_\alpha$$

Since K is a function of c only, and not a function of M or α, we may also write:

$$\mathcal{G} = \frac{ba^4S_n^2}{36}\left(\frac{dK}{dc}\right) \quad \text{.................................} (9)$$

Equation 9 gives the value of $\mathcal{G}$ for a particular set of experimental values of a, b, and (dK/dc). In solving fracture problems, one wishes a general formula for $\mathcal{G}$ in terms of the geometrical and loading conditions, and not involving the experimental quantity (dK/dc). In other words, what is desired is f in the expression:

$$\mathcal{G} = f(l, b, a, E, S_n, N)$$

The nature of f can be deduced by observing the effect of each of the variables l, b, a, E, S_n, and N on $\mathcal{G}$ in Eq 9, while varying them one at a time and holding the others constant.

Length does not appear directly in Eq 9, and (dK/dc) is not affected by length because $\Delta\theta$ does not depend on length, as shown by Fig. 17. Therefore, f will not depend on l.

The dependence of f upon b can be determined by applying elasticity theory to two beams, both having the same values of a, E, N, l, and S_n, but one having a width ($b_2 = mb_1$) that is m times the width b_1 of the other. The wide beam will have a bending moment that is m times that for the narrower beam, but $\Delta\theta$ will be the same for the two beams:

$$M_2 = mM_1$$

$$\Delta\theta_2 = \Delta\theta_1$$

Then:

$$K_2 = \frac{\Delta\theta_2}{M_2} = \frac{\Delta\theta_1}{mM_1} = \frac{1}{m} K_1$$

$$\left[\frac{dK}{dc}\right]_2 = \frac{1}{m}\left[\frac{dK}{dc}\right]_1$$

$$\mathcal{G}_2 = \frac{b_2 a^4 S_n^2}{36}\left[\frac{dK}{dc}\right]_2 = \frac{(mb_1)a^4 S_n^2}{36}\left\{\frac{1}{m}\left[\frac{dK}{dc}\right]_1\right\} = \mathcal{G}_1$$

which shows that f does not depend on b.

The dependence of f upon a can be determined in a similar manner by applying elasticity theory to two beams, both having the same values of N, E, and S_n, but one having a width, length, and net depth, each of which is n times the width, length, and net depth of the other. These two beams are geometrically similar, and so they will have the same $\Delta\theta$; but the bending moment of the second beam will be n^3 times as large as that of the first beam:

$$b_2 = nb_1$$

$$a_2 = na_2$$

$$l_2 = nl_1$$

$$\Delta\theta_1 = \Delta\theta_2$$

$$M_2 = n^3 M_1$$

Then:

$$K_2 = \frac{\Delta\theta_2}{M_2} = \frac{\Delta\theta_1}{n^3 M_1} = \frac{1}{n^3}(K_1)$$

$$(dK)_2 = \frac{1}{n^3}(dK)_1$$

Because the two beams are geometrically similar, it also follows that

$$c_2 = nc_1$$

$$(dc)_2 = n(dc)_1$$

Substituting the above values of $(dK)_2$ and $(dc)_2$:

$$\left[\frac{dK}{dc}\right]_2 = \frac{(dK)_2}{(dc)_2} = \frac{\frac{1}{n^3}(dK)_1}{n(dc)_1} = \frac{1}{n^4}\left[\frac{dK}{dc}\right]_1$$

$$\mathcal{G}_2 = \frac{b_2\, a_2^4 S_n^2}{36}\left[\frac{dK}{dc}\right]_2 = \frac{(nb_1)(na_1)^4 S_n^2}{36}\left\{\frac{1}{n^4}\left[\frac{dK}{dc}\right]_1\right\} = n\mathcal{G}_1$$

which shows that f contains a factor that is proportional to the first power of a.

The dependence of f upon E can be determined similarly by observing the effect of a change of modulus on each quantity in Eq 9. The only quantity affected by a modulus change is the numerator of the derivative, dK. Using p as the ratio of moduli for two beams of the same dimensions and loading:

$$E_2 = pE_1$$

$$M_2 = M_1$$

$$(\Delta\theta)_2 = \frac{(\Delta\theta)_1}{p}$$

$$K_2 = \frac{(\Delta\theta)_2}{M_2} = \frac{1}{p}\frac{(\Delta\theta)_1}{M_1} = \frac{1}{p}K_1$$

$$d(K_2) = \frac{1}{p}[d(K_1)]$$

we find that dK, and therefore also $\mathcal{G}$, varies inversely as the first power of E.

A similar analysis of the S_n-dependence of f, using q as the ratio of nominal stresses for two otherwise-identical beams, shows that:

$$(S_n)_2 = q(S_n)_1$$

$$M_2 = qM_1$$

$$(\Delta\theta)_2 = q(\Delta\theta)_1$$

$$K_2 = \frac{(\Delta\theta)_2}{M_2} = \frac{q(\Delta\theta_1)}{qM_1} = K_1$$

$$dK_2 = dK_1$$

$$dc_2 = dc_1$$

namely, that the derivative dK/dc in Eq 9 does not depend on S_n. Consequently $\mathcal{G}$ varies as the square of S_n.

The dependence of f on N is not easily determined by applying elasticity theory, and that is exactly why an experimental method is proposed. Consequently the dependence of f on N will be left in terms of an arbitrary function $F(N)$ for the time being and the experimental determination of this dependence will be explained presently. Summarizing:

$$\mathcal{G} = \frac{aS_n^2}{E}[F(N)] \quad \text{(10)}$$

By comparing Eqs 9 and 10, we see that the function $F(N)$ is related to the experimental conditions of a, b, and (dK/dc) in Eq 9 by:

$$F(N) = \frac{ba^3E}{36}\left(\frac{dK}{dc}\right) \quad \text{(11)}$$

and can be determined from the measurements in the manner indicated by the lower graph in Fig. 17.

APPENDIX IV

DERIVATION OF ENERGY RELEASE RATE FOR NOTCH TENSION IN TERMS OF LOAD-DEFLECTION CURVES FOR NOTCHED SPECIMENS

As with beams, we make load deflection curves for various notch depths. The deflection α is

$$\alpha = \Delta\sigma + \frac{Pl}{A_0E} \quad \text{(12)}$$

where P is load, l is gage length, A_0 is unnotched area, E is modulus of elasticity, Pl/A_0E is the deflection of the unnotched bar, and $\Delta\sigma$ is the extra deflection due to the notch. Again, we define K as the slope of the $\Delta\sigma$ *versus* P curve:

$$K = \frac{\Delta\sigma}{P}$$

Substituting into Eq 12:

$$\alpha = P\left(K + \frac{l}{A_0E}\right)$$

$$P = \frac{\alpha}{K + \dfrac{l}{A_0E}} \quad \text{(13)}$$

The energy release rate $\mathcal{G}$ is the amount of stored energy released for a small increase in crack area for a given total deflection α. If W is the stored energy for any notched area and A is the net cross-section of the bar, then $\mathcal{G}$ is:

$$\mathcal{G} = \left(\frac{-\,\partial W}{-\,\partial A}\right)_\alpha = \left(\frac{\partial W}{\partial A}\right)_\alpha \quad \text{(14)}$$

The stored energy at area A is:

$$W_A = \tfrac{1}{2}\,P\alpha$$

and at area $(A + dA)$ is:

$$W_{A+dA} = \tfrac{1}{2}\,\alpha\left[P + \left(\frac{\partial P}{\partial A}\right)_\alpha dA\right]$$

Taking the difference and substituting into Eq 14:

$$\mathcal{G} = \frac{\alpha}{2}\left(\frac{\partial P}{\partial A}\right)_\alpha \quad \text{(15)}$$

The derivative can be obtained by differentiating Eq 15. Noting that only K is a function of A, the derivative is:

$$\left(\frac{\partial P}{\partial A}\right)_\alpha = \frac{-\alpha\left(\dfrac{dK}{dA}\right)}{\left(K + \dfrac{l}{A_0E}\right)^2} \quad \text{(16)}$$

In this equation is not necessary to write dK/dA as a partial derivative with α constant because K is only a function of A. Substituting Eqs 13 and 16 into Eq 15:

$$\mathcal{G} = \frac{-\alpha^2 \left(\frac{dK}{dA}\right)}{2\left(K + \frac{l}{A_0E}\right)^2} = -\frac{P^2}{2}\frac{dK}{dA} \qquad (17)$$

Equation 17 will be more useful in terms of the nominal stress S_n:

$$S_n = \frac{P}{A} = \frac{4P}{\pi d^2} = \frac{4P}{\pi D^2(1 - N)} \qquad (18)$$

where D is unnotched diameter, d is notched diameter, and N is the fraction of area removed by the notch:

$$N = 1 - \frac{d^2}{D^2}$$

Solving Eq 18 for P and substituting into Eq 17 gives:

$$\mathcal{G} = -\frac{\pi^2 d^4 S_n^2}{32}\left(\frac{dK}{dA}\right) \qquad (19)$$

By methods similar to those used in Appendix III, it can be shown that $\mathcal{G}$ varies as the square of S_n, inversely as the modulus, and is independent of length. The dependence on N will be determined experimentally, while the dependence on d will be determined by application of elasticity theory, as follows. If a larger bar has an unnotched diameter $D_2 = nD_1$ that is n times larger than the unnotched diameter D_1 of a small bar, but has the same notch depth N, modulus E, and nominal stress S_n as the smaller bar, the other pertinent dimensions will have relative magnitudes as follows:

$$D_2 = nD_1$$

$$d_2 = nd_1$$

$$A_2 = n^2A_1$$

$$dA_2 = n^2(dA_1)$$

$$P_2 = n^2P_1$$

$$(\Delta\sigma)_2 = n(\Delta\sigma)_1$$

$$K_2 = K_1/n$$

$$dK_2 = dK_1/n$$

Substituting into Eq 19:

$$\mathcal{G}_2 = \frac{\pi^2 d_2^4 S_n^2}{32}\left(\frac{dK}{dA}\right)_2 = \frac{\pi(nd_1)^4 S_n^2}{32}\left(\frac{dK_1}{n}\right)\left(\frac{1}{n^2 dA_1}\right) = n\mathcal{G}_1$$

which shows that $\mathcal{G}$ varies as the first power of d. Thus:

$$\mathcal{G} = \frac{dS_n^2}{E}F(N) \doteq \frac{\pi^2 d^4 S_n^2}{32}\left(\frac{dK}{d\theta}\right)$$

where:

$$F(N) = -\frac{\pi^2 d^3 E}{32}\left(\frac{dK}{dA}\right)$$

DISCUSSION

Mr. G. R. Irwin[1] (*presented in written form*).—It was a pleasure to study the author's paper in which the analytical foundations for the measurements discussed are well stated and the experimental techniques are explained in useful detail.

In his Fig. 14 the author gives the experimental results for circumferentially notched round bars in tension. These appear to be high relative to theoretical calculations and the shape of the curve for the experimental results must be considered unlikely regardless of how well it represents the actual measured data. A number of investigators are using sharply notched round bars in tension to measure fracture toughness and it would be helpful if the uncertainties pertaining to a $\mathcal{G}$ value calculation, particularly at the 50 per cent netsection point could be reduced. The writer's 1955 suggestion of a shallow-notch equation, given as Eq 4 by the author, is not well founded. However, the writer has made estimates using Peterson's tables of stress concentration factors which essentially agree with Bueckner's calculations at an abscissa of 0.707 on the author's Fig. 14. This estimate is 18 per cent below the curve representing experimental results. The author states his experimental results were in general 40 per cent above theoretical estimates. There is no reason other than the greater experimental uncertainties pertaining to the round notched bar measurements why these experimental results should not agree with calculated values about as well as was found in the case of the notched bend tests. Thus the author's comment would be of interest on the point of whether the 18 per cent discrepancy referred to above is within the uncertainty range of the experimental measurements.

Attention is directed also to a discussion by the writer of a paper by Wundt[2] at the recent ASME Metals Engineering Division meeting at Albany and to the paper[3] by the writer at the same meeting. In the discussion a procedure for incorporating a correction for plastic strains is given. The effect of this would be to increase the $\mathcal{G}_c$ values given in the author's Table VI. In the writer's Albany paper a method is given for estimating a plane strain $\mathcal{G}_c$ value from a measurement performed using a plate of limited thickness. This procedure would lead to an estimated plane strain $\mathcal{G}_c$ value lower than the average value shown in the author's Table IV. When both factors are taken into account the discrepancy between the plane strain $\mathcal{G}_c$ values measured with round notched bars in tension and with notched bars in bending does not appear to be significant.

Mr. H. T. Corten.[4]—I would like to ask the author if he considered the state of stress at the root of the notches in the different specimens and the concept that Irwin is using of $\mathcal{G}_{Ic}$ for a plane strain condition as compared with $\mathcal{G}_c$ for a plane stress condition. Will this bring the values of $\mathcal{G}_c$ for different shape specimens into better agreement?

---

[1] Navy Department, Naval Research Laboratory, Washington, D. C.

[2] B. M. Wundt, "Unified Interpretation of Room-Temperature Strength of Notched Specimens as Influenced by Their Size," Am. Soc. Mechanical Engrs., Metals Engineering Conference, Albany, N. Y., April 29, 1959, Discussion by G. R. Irwin.

[3] G. R. Irwin, "Fracture Mode Transition for a Crack Traversing a Plate," Am. Soc. Mechanical Engrs., Metals Engineering Conference, Albany, N. Y., April 29, 1959.

[4] Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

MR. J. D. LUBAHN (*author*).—In answer to Mr. Corten's question Mr. Irwin has considered the effect of thickness of specimen, that is, the range of conditions between plain strain and plain stress. We know that the apparent value of $\mathcal{G}_c$ that you will get increases considerably as the thickness goes down in the range of thicknesses corresponding to "sheet." All of the tests that I have described are for quite chunky things, and so they represent essentially plain strain. The analytical work is for the same condition, plain strain. How you need to modify these concepts as you approach plain stress I do not know, but I am hoping that Mr. Irwin will find out for us.

I want to thank Mr. Irwin for his comments. I am sure that additional work will close the gap between experimental and analytical results. Certainly there is room for improvement in the accuracy of the experimental work, especially that pertaining to notch tension testing.

EFFECT OF COMPOSITION AND PROCESSING VARIABLES ON SOME MECHANICAL PROPERTIES OF LARGE NICKEL-MOLYBDENUM-VANADIUM ROTOR FORGINGS*

BY K. H. KRAMER AND J. E. STEINER[1]

SYNOPSIS

A multiple regression analysis has been made to estimate the coefficients in equations relating the impact and tensile properties of large nickel-molybdenum-vanadium turbine and generator forgings to composition and processing variables. The analysis indicated that the fracture-transition temperature increases with increased carbon, manganese, molybdenum, and phosphorus contents, and austenitizing temperature, and that it decreases with increased nickel content and tempering temperature. The analysis also indicated that the tensile strength and yield strength increase with increased carbon, nickel, chromium, and molybdenum contents, and decrease with increased tempering temperature. These results are expressed quantitatively in the form of equations. The most important conclusion from this analysis is that both impact and tensile properties of large Ni-Mo-V steel forgings should be improved by increasing the nickel content of the steel.

The several catastrophic failures of large turbine and generator rotor shafts that occurred in the United States during the past several years have caused considerable concern among both the producers of the forgings from which the shafts are made and the electrical equipment manufacturers who purchase and use these shafts.[2] Efforts to determine the cause of these failures have, in practically every instance, revealed that the steel failed in a brittle manner and that the impact transition temperatures of the steel fragments were high. Accordingly, intensive research programs aimed at developing steels with improved notch toughness have been undertaken by both producers and users of large forgings.

As one approach to obtaining improved notch toughness in rotor forgings, several forging producers and users have applied statistical methods to determine the coefficients in equations relating the V-notch Charpy fracture-appearance transition temperatures of large nickel-molybdenum-vanadium forgings to composition and processing variables. The present paper describes such an approach by the U. S. Steel Applied Research Laboratory, in which a regression analysis was made to solve the above problem. Also, equations relating tensile strength and yield strength to composition and processing variables were computed.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] U. S. Steel Corp., Applied Research Laboratory, Monroeville, Pa.

[2] E. E. Thum, "Recent Accidents with Large Forgings," *Metal Progress*, Feb., 1956.

DATA

The data used in the present study were obtained from melting, ladle-analyses, and inspection records of 173 commercial Ni-Mo-V turbine and generator rotor forgings produced by several manufacturers. The composition variables considered were carbon, manganese, phosphorus, sulfur, silicon, nickel, chromium, molybdenum, and vanadium contents. Processing variables considered were melting practice (electric furnace, basic open-hearth, or acid open-hearth), treating diameter, austenitizing temperature, austenitizing time, the lowest temperature to which the forging was cooled after austenitizing, equalizing temperature after air cooling from the austenitizing temperature, type of cooling (fan or air), tempering temperature, and producers. Data were supplied by U. S. Steel and three other producers that we shall call producer A, producer B, and producer C. All the specimens for the impact and tension tests were obtained so that they were oriented in the radial direction and represented the steel near the surface of the forgings (one end of each specimen represented the surface). It is, therefore, not to be expected that the resulting equations will necessarily apply to the steel from the interior portions of the forgings (for example, at the mid-radius or the axial center positions).

The mechanical properties considered in this study were Charpy V-notch fracture-appearance transition temperatures, tensile strengths, and yield strengths (0.02 per cent offset).

ANALYSIS

It was desired to obtain an equation of the form:

Transition temperature $= a_0 + a_1 \times$ per cent carbon $+ a_2 \times$ per cent manganese $+ a_3 \times$ per cent phosphorus $+ a_4 \times$ per cent sulfur $+ a_5 \times$ per cent silicon $+ a_6 \times$ per cent nickel $+ a_7 \times$ per cent chromium $+ a_8 \times$ per cent molybdenum $+ a_9 \times$ per cent vanadium $+ a_{10} \times$ austenitizing temperature $+ a_{11} \times$ austenitizing time $+ a_{12} \times$ tempering temperature $+ a_{13} \times$ the lowest temperature to which the forging is cooled after austenitizing $+ a_{14} \times$ treating diameter $+ a_{15} \times$ equalizing temperature after final air cool.................... (1)

where the value of a_0 depends on the melting practice (electric furnace, basic open-hearth, or acid open-hearth), the type of cooling (fan or air), and the producer. It was also desired to obtain similar equations for tensile strength and for yield strength.

Data on all these composition and processing variables were not included for calculating the equations, however, except for the forgings produced by U. S. Steel. In particular, the austenitizing times, the types of cooling, the lowest temperatures to which the forgings were cooled after austenitizing, and the equalizing temperatures after final air cool were not supplied by producers A, B, or C. Therefore, these variables were omitted from the equations that were calculated with the data supplied by all four producers. Austenitizing temperatures were also omitted because there was insufficient variation in austenitizing temperature among the forgings of any one of the producers except U. S. Steel.

Multiple linear regression equations for transition temperature, tensile strength, and yield strength were calculated using all the remaining variables in Eq 1. The equation for transition temperature explained about 71 per cent of the over-all variation in transition temperature, the equation for tensile strength explained about 68 per cent of the over-all variation in tensile strength, and the equation for yield strength explained about 60 per cent of the over-all variation in yield strength. Examination of all the results at this point indicated that only the variables carbon, manganese, phosphorus, molybdenum, and nickel content, tempering temperature, and producer had a real influence on the transition temperature, and that only carbon, molybdenum, nickel, and chromium contents, tempering temperature, and producer had a real influence on the tensile strength and the yield strength. With only these variables, new equations for transition temperature, yield strength, and tensile strength were computed as follows:

Transition temperature $F = a + 300 \times$ per cent carbon $+ 120 \times$ per cent manganese $+ 1700 \times$ per cent phosphorus $- 17 \times$ per cent nickel $+ 150 \times$ per cent molybdenum $- 0.18 \times$ tempering temperature, deg Fahr. . . . . (2)

where $a = 128$ for producers A, B, and C, and where $a = 167$ for U. S. Steel.

Tensile strength, 1000 psi $= a + 62 \times$ per cent carbon $+ 11 \times$ per cent nickel $+ 18$ per cent chromium $+ 51 \times$ per cent molybdenum $- 0.13 \times$ tempering temperature, deg Fahr. . . . . (3)

where $a = 184$ for producer C and $a = 176$ for the other producers.

Yield strength, 1000 psi $= a + 25 \times$ per cent carbon $+ 15 \times$ per cent nickel $+ 16 \times$ per cent chromium $+ 52 \times$ per cent molybdenum $-0.09 \times$ tempering temperature, deg Fahr. . . . . (4)

where $a = 119$ for producer C, and $a = 112$ for the other producers.

Equation 2 explains about 68 per cent of the over-all variation in the transition temperature and has a standard error of estimate of 18 F; Eq 3 explains 66 per cent of the over-all variation in tensile strength and has a standard error of estimate of 4500 psi; and Eq 4 explains 58 per cent of the over-all variation in yield strength and has a standard error of estimate of 5100 psi.

Equations were also calculated with only the U. S. Steel data. The coefficients in these equations, in general, differed from the corresponding coefficients calculated with all the data only by amounts within the limits of the accuracy of these coefficients. As mentioned above, U. S. Steel's data included many processing variables that were not included in the data furnished by the other producers. Of these variables, only austenitizing temperature was found to have a real influence on the transition temperature of the forgings, while none of these variables was found to have any real influence on the tensile strength or the yield strength. This estimated effect of austenitizing temperature can be used to improve Eq 2, at least so far as U. S. Steel's forgings are concerned. The resulting equation is as follows:

Transition temperature (deg Fahr) $= -300 + 300 \times$ per cent carbon $+ 120 \times$ per cent manganese $+ 1700 \times$ per cent phosphorus $- 17 \times$ per cent nickel $+ 150 \times$ per cent molybdenum $- 0.18 \times$ tempering temperature (deg Fahr) $+ 0.31 \times$ austenitizing temperature. . . . . (5)

This equation applies only to U. S. Steel's forgings.

Table I gives the important regression coefficients of the transition temperature, tensile strength, and yield strength equations. The regression coefficient, in each case, indicates the change in the transition temperature, tensile strength, or yield strength corresponding to a unit change (for example, 1 per cent in composition, or 1 F in temperature) in the independent variable.

Figure 1 illustrates the agreement of observed with predicted transition temperatures, tensile strengths, and yield strengths for the 99 U. S. Steel forgings from which the data were obtained for the present analysis. Notice that for each property approximately 95 per cent of the points fall within two standard errors of the predicted values.

TABLE I.—IMPORTANT REGRESSION COEFFICIENTS.

Independent Variable	Regression Coefficients and Their 95 per cent Confidence Intervals			Range of Independent Variable
	Transition Temperature, deg Fahr	Tensile Strength, 1000 psi	Yield Strength, 1000 psi	
Molybdenum, per cent	150 ± 40	51 ± 10	52 ± 11	0.25 to 0.61
Manganese, per cent	120 ± 50	....	....	0.47 to 0.76
Carbon, per cent	300 ± 140	62 ± 34	25 ± 38	0.19 to 0.30
Nickel, per cent	−17 ± 12	11 ± 3	15 ± 4	1.90 to 3.00
Chromium, per cent	....	18 ± 8	16 ± 9	0.02 to 0.50
Phosphorus, per cent	1700 ± 500	....	....	0.006 to 0.033
Austenitizing temperature, deg Fahr	+0.31 ± 0.18	....	....	1450 to 1562
Tempering temperature, deg Fahr	−0.18 ± 0.11	−0.13 ± 0.03	−0.09 ± 0.03	1060 to 1220
Producer, U. S. Steel *versus* others	39 ± 8	....	....	....
Producer C *versus* others	....	7 ± 4	8 ± 4	....
Standard error	18	4.5	5.0	....
Multiple correlation coefficient	0.82	0.81	0.76	....

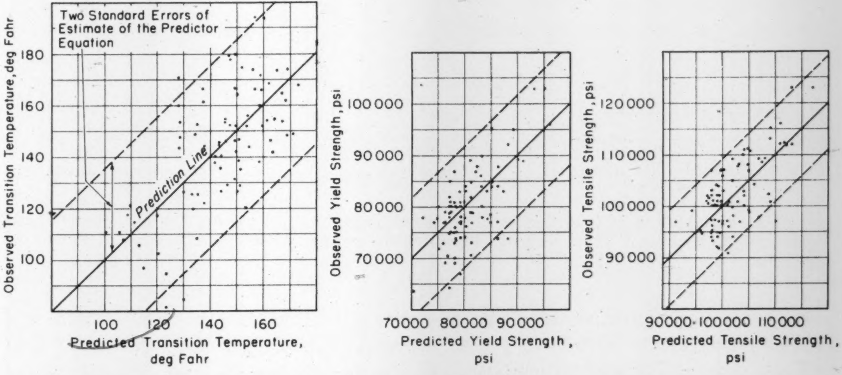


FIG. 1.—Comparison of Observed with Predicted Transition Temperature, Yield Strength, and Tensile Strength of U. S. Steel Forgings Used to Compute the Predicting Equations.

APPLICATION TO RECENTLY PRODUCED FORGINGS

Partly as a result of this analysis, some changes were made in the aim composition and in the heat-treating practices employed in producing U. S. Steel Ni-Mo-V forgings. As a consequence, the average transition temperature decreased from 143 F for the 99 forgings studied in the regression analysis to 120 F for the next 25 forgings produced after the regression analysis was done. Concurrently, the average tensile strength increased from 102,500 to 106,000 psi and the average yield strength increased from 80,600 to 81,900 psi.

The composition changes effected, in terms of average per cent, were a decrease in manganese content from 0.59 to 0.53 per cent, a decrease in phosphorus content from 0.012 to 0.009 per cent, an increase in nickel content from 2.73 to 2.85 per cent, an increase in chromium content from 0.12 to 0.28 per cent, and a decrease in molybdenum content from 0.48 to 0.32 per cent. There was no appreciable change in carbon content. The average austenitizing temperature was increased from 1483 to 1514 F, and the average tempering temperature was decreased from 1151 to 1090 F.

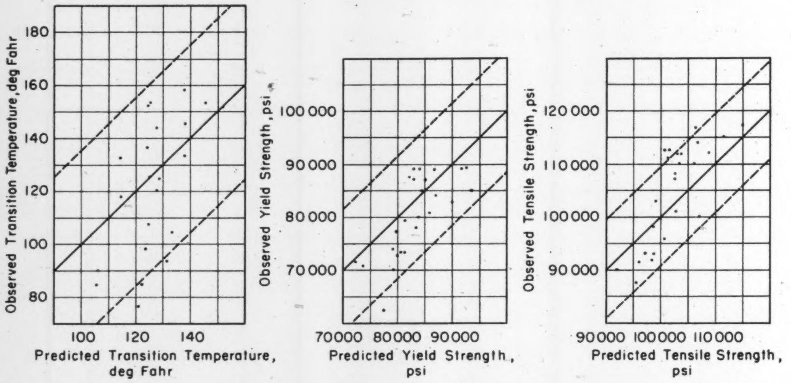


FIG. 2.—Comparison of Observed with Predicted Transition Temperature, Yield Strength, and Tensile Strength of U. S. Steel Forgings Produced Subsequent to the Computation of Predicting Equations.

These changes in chemical composition and heat-treating practice, according to Eqs 3, 4, and 5, should have resulted in a decrease of 19 F in the average transition temperature, an increase of 4100 psi in the average tensile strength, and an increase of 1700 psi in the yield strength. Notice that these predicted changes are reasonably close to the changes actually obtained.

Figure 2 illustrates the comparison of the actual with predicted transition temperatures, tensile strengths, and yield strengths of the 25 forgings produced after the regression analysis was done. Although most of the points lie within the two-standard-error prediction band, the fit of these data to the prediction line is not so good as the fit of the original data, as shown in Fig. 1. In view of the substantial changes that were made in the chemical composition and the heat-treating practice for the forgings represented in Fig. 2, the fit observed is considered reasonable. Also, the fit of these data suggests that, as the composition and processing of successive groups of production forgings changes, it would be desirable to recompute periodically the prediction equations with data from a sufficient sample of recent forgings.

At present, the chemical composition and the heat-treating practices employed by U. S. Steel in the manufacture of large Ni-Mo-V rotor forgings are gradually being modified in the directions suggested by the present regression analyses and other research programs. The nickel content of the Ni-Mo-V steel, for example, will undoubtedly be further increased to levels substantially higher than those mentioned in the present study. Thus, with information from the periodic computation of new prediction equations to supplement other experimental results, it is expected that continued improvements in the notch toughness, yield strength and tensile strength of large Ni-Mo-V forgings will be accomplished.

Acknowledgment:

The authors express their appreciation to the Materials and Processes Laboratory of the Large Steam Turbine and Generator Department, General Electric Company, who furnished much of the data and to the Metallurgical Department of the Homestead District Works of U. S. Steel Corporation for providing additional data.

BEHAVIOR OF DUCTILE CAST IRON UNDER COMPRESSIVE STRESS

By Paul K. Trojan,[1] R. A. Flinn,[1] and D. J. Reese[2]

Synopsis

The purpose of this investigation was to study the behavior of various ductile irons under compressive stress.

Compression specimens were machined from ½, 1, and 2 in. "Y" blocks; austenitized and quenched from 1600 F and tempered 1 hr at 1300, 1150, 925, and 300 F. Compression yield strength for 0.1 per cent offset ranged from 66,000 psi (215 Brinell hardness number (BHN)) to 215,000 psi (585 BHN). Within this range the maximum deviation among the three section sizes was only ±4000 psi and ±8 BHN. However the compression modulus did indicate some sensitivity to the less perfect graphite spheroids present in the 1-, and 2-in. sections. At low hardness levels the larger sections gave a compression modulus of 4×10^6 psi lower while at higher hardness the variation was 2×10^6 psi. The over - all average modulus was 24×10^6 psi.

Compression data of metallic materials are far less common in the literature even though important in bearing stresses and compressive surface wear problems. In many design problems, hardness and tension data are used as an index of possible service performance under compressive loading, while actual compression data have been a neglected determination.

In a gray cast iron, mechanical properties are known to change with graphite size, shape, and distribution. This is largely due to the presence of the inherent micro-notch characteristics of flake graphite. In ductile iron, however, the sharp notch problem is not present when spheroidal graphite is formed; therefore it might be expected that most physical properties would approach those of steel. There should therefore be a similarity in compression data between ductile iron and steel.

Procedure

A ductile cast iron containing 3.60 per cent carbon, 2.25 per cent silicon, 0.54 per cent manganese, 1.04 per cent nickel, and 0.072 per cent magnesium was cast in ½-, 1-, and 2-in. "Y" block molds using accepted basic induction furnace practice. The graphite characteristics are illustrated in Fig. 1.

Specimens nominally 2½ in. long by ⅞ in. in diameter were machined from the 1 and 2 in. "Y" blocks. These were austenitized at 1600 F, quenched in oil, and three specimens from each "Y" block section were tempered 1 hr at each of the following temperatures: 300, 925, 1150, and 1300 F and water quenched. The tempering procedure gave a matrix which

---

[1] Cast Metals Laboratory, Department of Chemical and Metallurgical Engineering, University of Michigan, Ann Arbor, Mich.

[2] Director, Ductile Iron Division International Nickel Co., Inc., New York, N. Y.

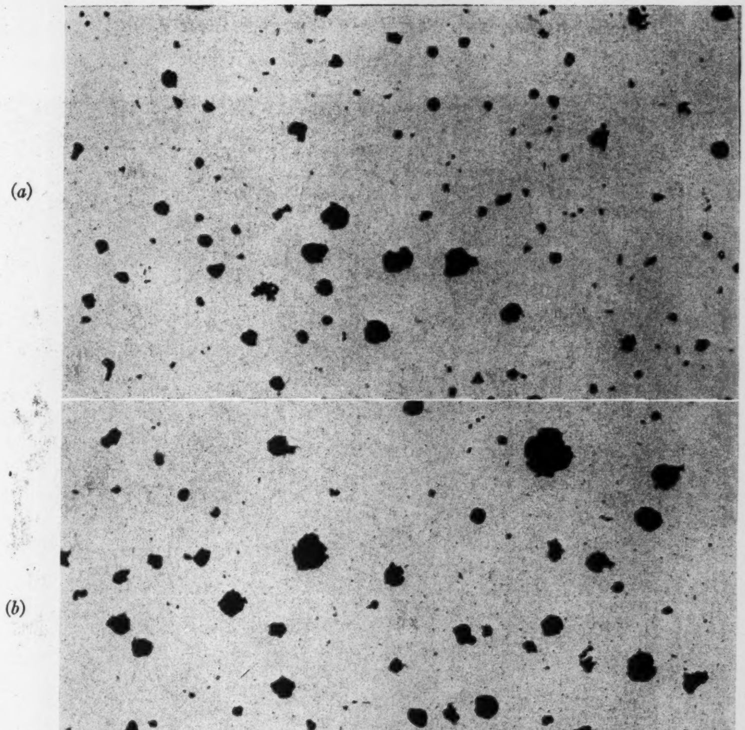


FIG. 1. Microstructure of Graphite (× 100).

(a) Typical spheroidal from the ½-in. "Y" block. Unetched.
(b) Spheroidal from 1-in. "Y" block. Unetched.
(c) In 2-in. "Y" block. Unetched. Note presence of some less perfect spheroids.

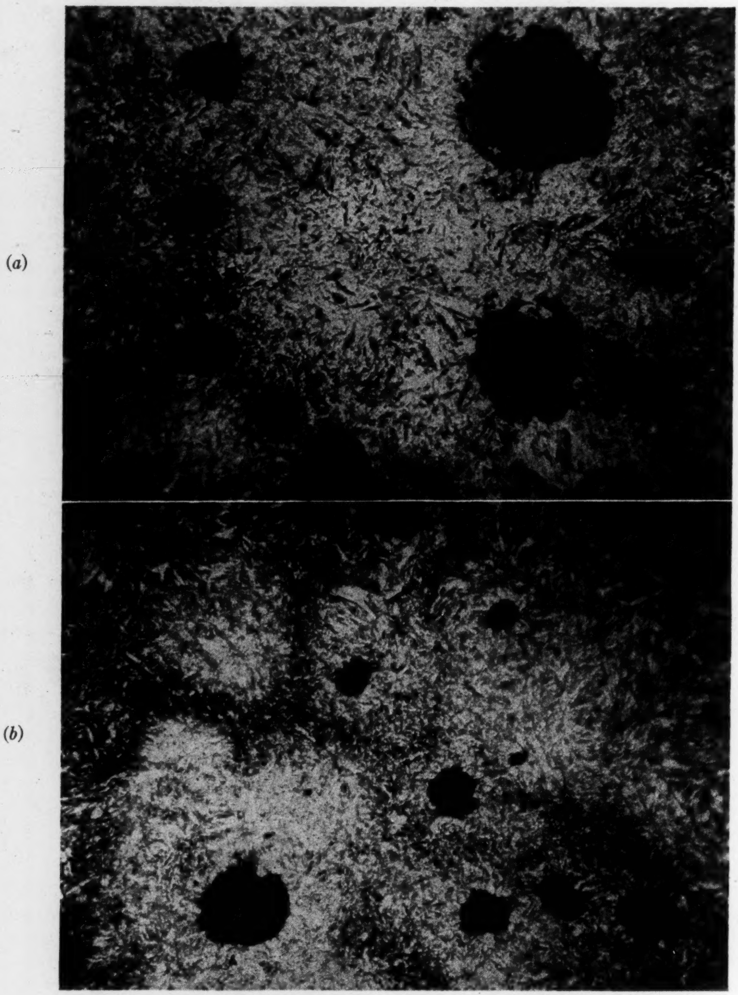


FIG. 2.—½-in. "Y" Block. (× 500).
(*a*) Tempered 1 hr at 300 F, Brinell hardness number 580. Nital etch. Martensite is slightly tempered and some retained austenite remains.
(*b*) Tempered 1 hr at 925 F, Brinell hardness number 365. Nital etch. Tempered martensite has more feathery appearance.

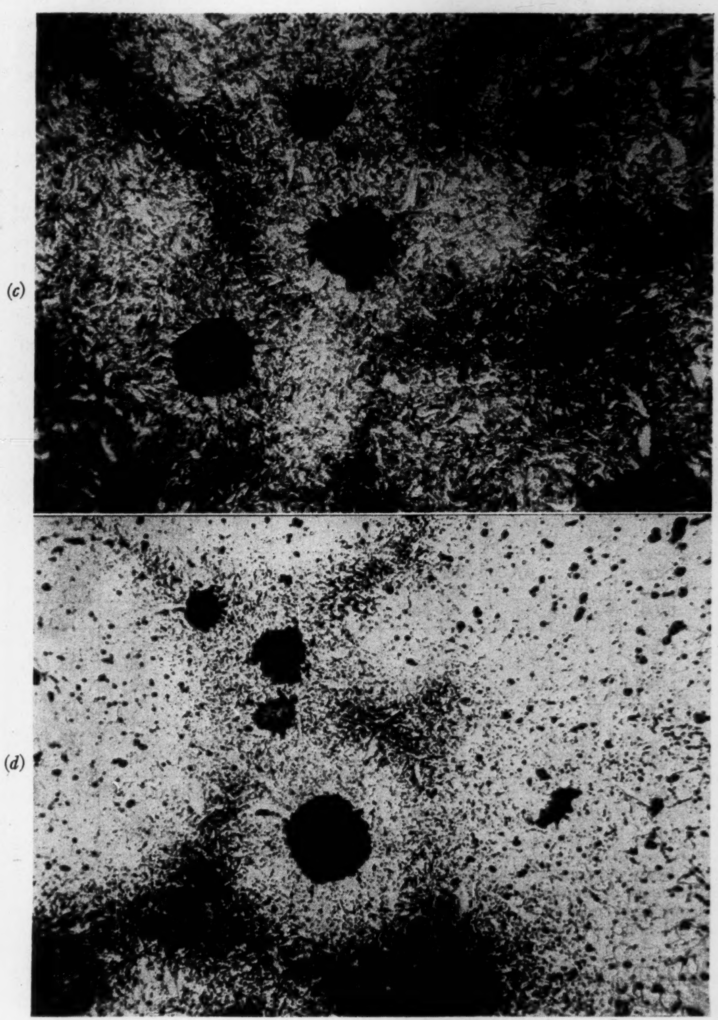


FIG. 2.—*Concluded.*

(*c*) Tempered 1 hr at 1150 F, Brinell hardness number 285. Nital etch. Beginning of spheroidization.

(*d*) Tempered 1 hr at 1300 F, Brinell hardness number 215. Nital etch. Spheroidization is almost complete with some secondary precipitation of graphite.

FIG. 3.—Compression Specimen From 1-in. "Y" Block. Approximately full size (2.375 in. long by 0.798 in. in diameter).

Two SR-4 strain gages are in place on opposite sides and are connected in series.

TABLE I.—RESULTS OF COMPRESSION AND HARDNESS STUDY.

"Y" Block Section Size, in.	Heat Treatment, 1 hr Temper, deg Fahr	Compression Modulus, psi[a]	Compression Yield, psi[a]		Hardness		Tensile Strength, psi[b]
			0.1 per cent offset	0.2 per cent offset	Rockwell C Scale	Brinell Hardness Number—Tungsten Carbide Ball	
½..........	1300	25×10^6	62 000	73 000	17.7	213	101 000
1...........	1300	21.5	69 500	75 500	16.1	219	
2...........	1300	20.5	66 500	73 250	15.3	216	
½..........	1150	27	112 000	117 000	30.3	284	138 000
1...........	1150	23.5	107 500	114 500	30.8	298	
2...........	1150	23.75	110 500	116 500	30.5	291	
½..........	925	25.5	151 000	159 000	40.0	364	178 000
1...........	925	23.75	154 000	161 500	40.4	376	
2...........	925	23.75	152 000	159 500	39.6	380	
½..........	300	25.5	218 000	270 000	57.5	580	[c]
1...........	300	23.5	210 000	250 000	57.9	583	
2...........	300	23.5	215 000	260 000	58.3	592	

[a] Average of triplicate determinations.
Strain measurements by duplicate SR-4 gages.
[b] Curve for quench and tempered ductile iron (**3**).
[c] *K* curve does not hold for these high Brinell hardness numbers.

ranged from a lightly tempered martensite to an almost completely spheroidized structure as shown in Fig. 2. Each of the 24 specimens from these two section sizes was then remachined in accordance with ASTM Method A 256 – 46 to 2.375 in. long by 0.798 ± 0.001 in. in diameter.[3] Due to size limitations in the ½-in. "Y" block, specimens were machined to one half size as specified in Method A 256 – 46. The length to diameter ratio of 3 to 1 was maintained. Heat treatment was the same as for the larger samples.

Two SR-4 strain gages with the same gage factor were applied to opposite sides of each specimen and connected in series (Fig. 3). This had the effect of averaging the strain readings in the event of slight eccentric loading (**2**).[4] The small specimens had gages with a gage length of ¼-in., while in the larger sample gages with $\frac{13}{16}$ in. gage lengths were employed.

In testing, strain readings were taken every 2000 psi for specimens of low hardness and every 4000 psi at higher hardness levels. Readings were generally taken until plastic strain of 100 to 200 microinches occurred for every 1000 psi increase. This normally took place near the limit of calibration of the gage.

---

[3] Standard Method of Compression Testing of Cast Iron (A 256 – 46), 1958 Book of ASTM Standards, Part 3, p. 8.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

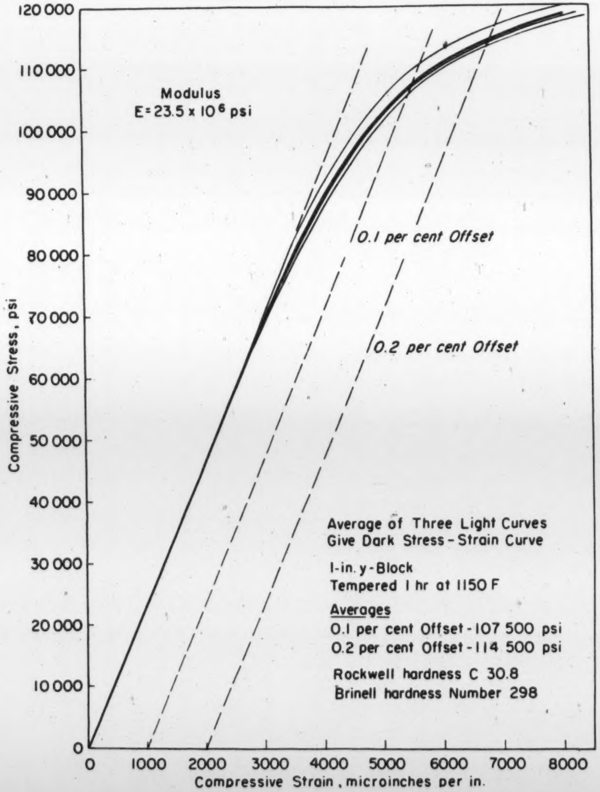


FIG. 4.—Typical Stress-Strain Curve for Ductile Iron in Compression.

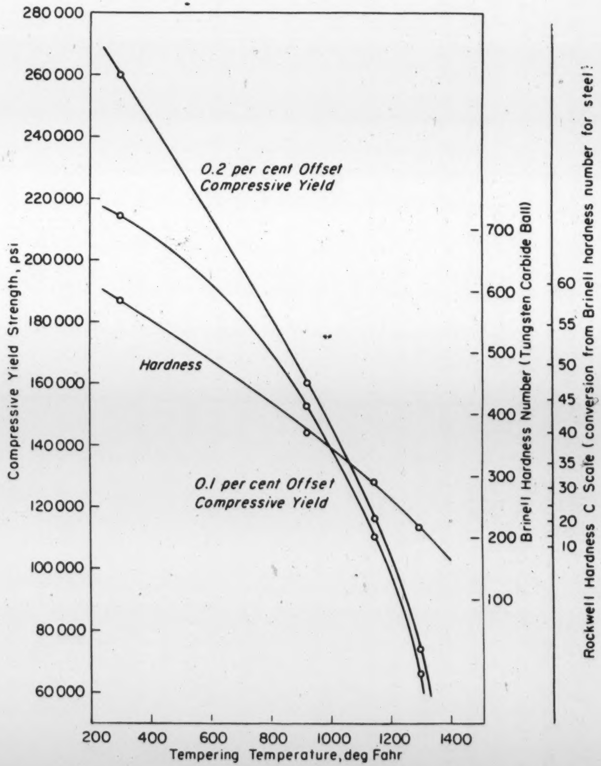


FIG. 5.—Compressive Yield Strength and Hardness as a Function of Tempering for 1 hr.

TEST RESULTS AND DISCUSSION

Results of the compression and hardness study are summarized in Table I. Tensile data are also included for comparison (3). Since three specimens from each heat treatment and section size were run, the tabulated stress values represent an average of three separate stress-strain curves to obtain one curve. A typical set of curves is shown in Fig. 4. The Rockwell hardness values represent an average of 9 values; the Brinell hardnesses an average of 6 values. The average data from Table I are reproduced in Figs. 5 and 6 which indicate the graphical relationship between the variables. The Rockwell hardness C Scale coordinate in these figures is obtained by conversion to the Brinell hardness numbers for steel. The conversion seems justified since, when the average Rockwell hardness C Scale for three section sizes at a given tempering temperature is converted to a Brinell hardness number; the difference between this number and the average experimental Brinell hardness number is only ±4 BHN at all four tempering temperatures.

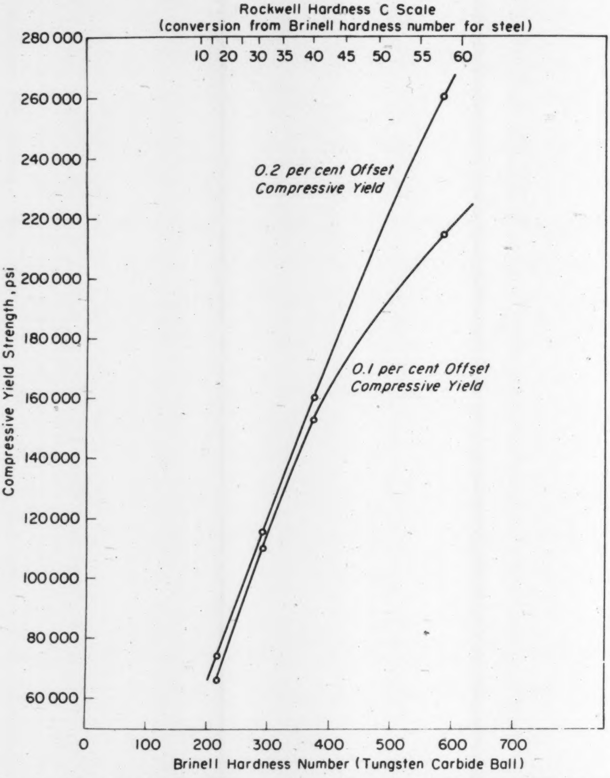


FIG. 6.—Compressive Yield Strength as a Function of Tempered Hardness.

It will be noted that both 0.1 and 0.2 per cent offset compressive yield strengths are recorded. In cast irons the 0.2 per cent offset is the normal one of interest and is generally not far removed from the 0.1 per cent offset at lower hardness levels. However at the higher hardness levels apparent work hardening is more severe. The net result is a range of almost 45,000 psi between the two offset methods at 585 BHN. Since low tempering temperatures still result in some retained austenite, the apparent high degree of work hardening might be partially attributed to the transformation to martensite as induced by the high stress levels required to produce yielding. This dependence of yield strength upon choice of per cent offset suggests 0.1 per cent offset might be a more conservative approach to yielding at these high hardness levels when severe plastic deformation is to be avoided.

Specimens were not run beyond the limits of the strain gage, a stress well below that required to cause failure. After yielding the sample begins to "barrel"—in most cases unsymmetrically. This is primarily due to the end restraint imposed by friction with the test head. Failure is similar to that found in any column. Therefore, since the load can no longer be considered as uniaxial after appreciable yielding has occurred, the recording of an ultimate compression strength would be a meaningless value.

Figure 6 indicates an approximate linearity between hardness and tempering temperature which normally does not exist beyond 1100 F (3). However it should be appreciated that 1 hr at the tempering temperature does not homogenize the matrix and does not cause secondary graphite precipitation sufficient to change the slope of the hardness curve at 1100 F.

The nature of the compressive yield curve is quite similar to that found in alloy steels. In tension, the yield strength of ductile iron is 10 to 15 per cent lower than that of steel, which is approximately the same relative position in compression yielding of these two materials within the temperature range of 700 to 1000 F tempering temperature (1). Within the section size limitations, a change in graphite characteristics has no significant effect on compressive yield strength. Therefore, the compressive yield strength of ductile cast iron is primarily dependent upon the matrix, since spheroidal graphite provides little effect of micro-notches as compared to flake graphite, where the matrix can exhibit less of its quality (4).

However, a change in graphite characteristics does change the compression modulus slightly as seen in Table I. It should be noted however that the variation due to the spheroidal graphite in larger sections is less at the higher hardness level. The same phenomenon is encountered in gray cast irons where flake graphite has a lesser detrimental effect on the modulus in an acicular matrix which has only been lightly tempered.

CONCLUSIONS

The compressive yield strength of ductile iron is independent of section size from $\frac{1}{2}$ to 2-in. provided the matrix is kept constant. There is, however, a slight difference in the compression modulus.

REFERENCES

(1) J. V. Melonas and J. R. Kattus, "Determination of Tensile, Compressive, Bearing, and Shearing Properties of Ferrous and Non-Ferrous Structural Sheet Metals at Elevated Temperatures," *Technical Report 56-340*, Wright Air Development Center, Oct. 1956.

(2) C. C. Perry and H. R. Lissner, "The Strain Gage Primer," McGraw-Hill Book Co., Inc., New York, N. Y. (1955).

(3) Metals Handbook Supplement, "Nodular Cast Iron," Am. Soc. for Metals, ASM Committee on Nodular Cast Iron, July 1954.

(4) R. A. Flinn and P. K. Trojan, "Simple Equipment Opens Research Door," *Modern Castings*, Sept. 1955.

THE ANNEALING BEHAVIOR OF 70-30 BRASS STRIP (CARTRIDGE BRASS, 70 PER CENT)

By Verne Pulsifer[1]

SYNOPSIS

Diagrams are presented showing the grain size in 70-30 brass (cartridge brass, 70 per cent) as a function of the four primary variables of time, temperature, cold reduction, and prior grain size. Information is also included on the activation energy for grain growth and the relationships between prior grain size, the origin of nuclei, and the origin of annealing textures.

While the salient features covering the annealing process of brass have been presented in considerable detail, a unified description is lacking. To give a broad view of the subject, a number of observations are made herein, which may be of widespread interest. The new aspects are presented with considerable reliance placed upon published results, and new data are presented only where it appears necessary to reinforce the conclusions.

The principal results are given in Figs. 1 and 2 showing the grain size of 70-30 brass as a function of the four primary variables of time, temperature, cold reduction, and prior grain size. These principal results are discussed in the first section of the paper. This is followed by sections dealing with (*a*) the experimental work and how it was used, (*b*) a discussion of additional results relating to grain growth, and (*c*) a discussion of preferred orientation.

VARIABLE FACTORS AFFECTING GRAIN SIZE

Figure 1 shows the results for a constant prior grain size of 0.530 mm, and Fig. 2 for an 0.053 mm prior grain size. A third figure, not shown but easily visualized, consists of the vertical projection of the values given in the base plane of Figs. 1 and 2. In other words, with an extremely fine prior grain size, the final grain size is independent of the final rolling and annealing treatment.

It will be observed from Figs. 1 and 2 (and the imaginary one) that the combined effect of increased prior grain size and decreased final reduction is that the grain growth values fan out toward smaller values of annealing time and temperature. This is consistent with well-known behavior, but it has not been given previously in a quantitative form. Intermediate prior grain sizes will give results in corresponding intermediate positions between those shown. The results cover the range of grain sizes from 0.002 mm to over 0.500 mm, a range greater than the usual annealing range used in commercial work. Each point in the volume defined in Figs. 1 and 2 (and implicit for all immediate prior grain sizes) is a single valued point. The space region can be imagined to consist of planes representing constant grain sizes.

It is possible to use such figures with confidence because of the relatively few

---

[1] Technical Administrator, Fansteel Metallurgical Corp., North Chicago, Ill.; formerly associated with Armour Research Foundation of Illinois Institute of Technology.

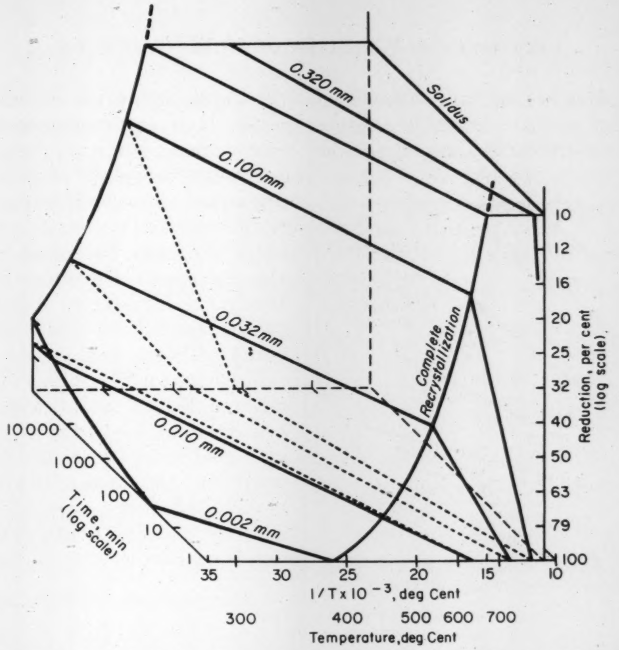


FIG. 1.—Complete Final Grain Size Figure for 0.530 mm Prior Grain Size in 70-30 Brass Strip.

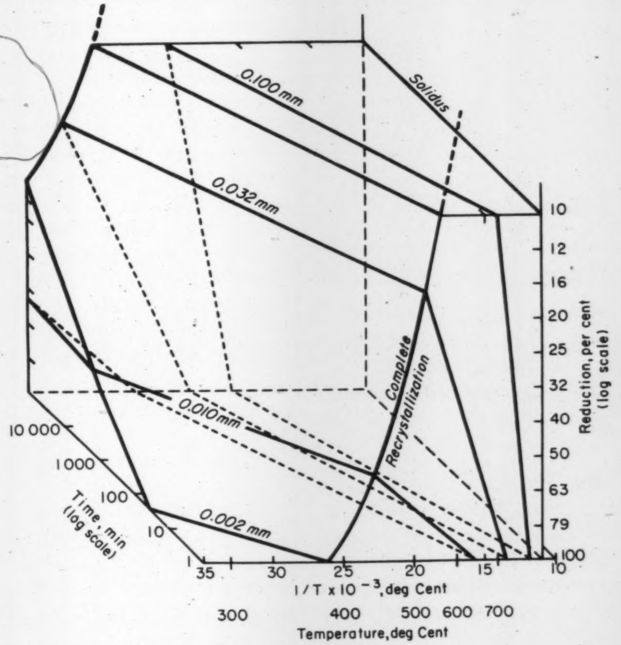


FIG. 2.—Complete Final Grain Size Figure for 0.053 mm Prior Grain Size in 70-30 Brass Strip.

aberrating factors that have to be taken into account in 70-30 brass. Aberrating influences are primarily those of (*a*) the retarding effect on grain growth of the strip surfaces when the grain size approaches one-tenth to one-third the strip thickness and (*b*) the effect of chemical impurities or variations in composition which occur unintentionally and technically disqualify the material as 70-30 brass. It is an interesting observation that during casting, the zinc ebullition normally cleanses the alloy of suspended impurities, dissolved gases, and more readily oxidized metals. As a result, 70-30 brass is usually free of such impurities above a certain level of concentration, so that grain size in brass is observed to be widely reproducible under normal processing circumstances.

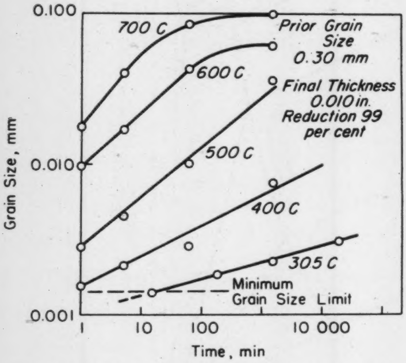


FIG. 3.—Effect of Time of Anneal on Grain Size of Brass Strip After 99 per cent Cold Reduction.

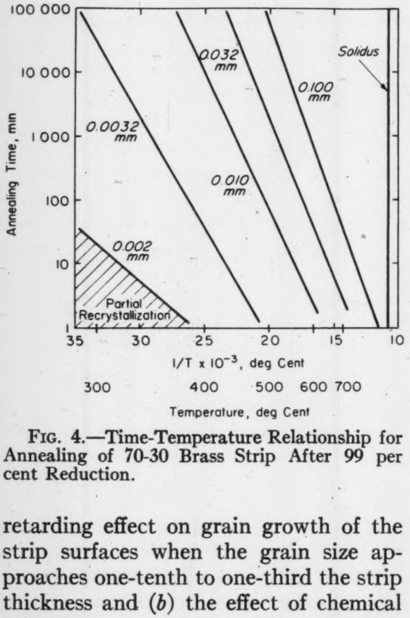


FIG. 4.—Time-Temperature Relationship for Annealing of 70-30 Brass Strip After 99 per cent Reduction.

The basis for Figs. 1 and 2 is a development of Figs. 3 and 4. Figure 3 shows new data obtained from annealing brass strip cold rolled 99 per cent from a prior grain size of 0.300 mm. It will be shown, however, that the prior grain size has no effect on the final grain size after such severe cold work.

EXPERIMENTAL PROCEDURE AND DISCUSSION OF RESULTS

The following section gives a description of the experimental work and a discussion of effects relating to the results from these experiments and how they relate to Figs. 1 and 2.

The brass alloy used for the data in Fig. 3 was cast from electrolytic copper and zinc, and the ingot analyzed 70.6 per cent copper. The ingot was hot-forged to 1 by 6 by 12 in. and cold rolled to 0.010 in. thickness. The strip was trimmed as rolling progressed to minimize edge-cracking, with a final width of 3½ in. obtained. Grain size determinations after annealing were made on cross-sections of the strip. The grain size distribution was uniform through the sections except after the high-temperature anneals.

Three features of the data given in Fig. 3 are significant at this point. First, the linearity of the log-log plot is lost at grain sizes above about 0.075 mm diameter. This corresponds to 0.003 in., or about 3½ grain diameters across the thickness of the strip. Walker and Beck

(1)[2] have shown that the grain growth in brass is restricted as the strip thickness is approached. On this particular observation in the ordinary annealing of brass there is little disagreement, and the present work confirms this view. Therefore, any quantitative use that is made of Figs. 1 and 2 requires a special consideration of the ratio between strip thickness and grain size.

The second feature is in regard to the initial temperatures at which recrystallization occurs. It is estimated by the presentation of the data in Fig. 3 that recrystallization might be initiated at a temperature as low as 250 C. The time required for this event would be extremely long but would be consistent with Averbach's finding (2) that the minimum recrystallization temperature occurs near 250 C.[3] A horizontal line shows the minimum grain size obtained at 99 per cent reduction, a value which is independent of time and temperature.

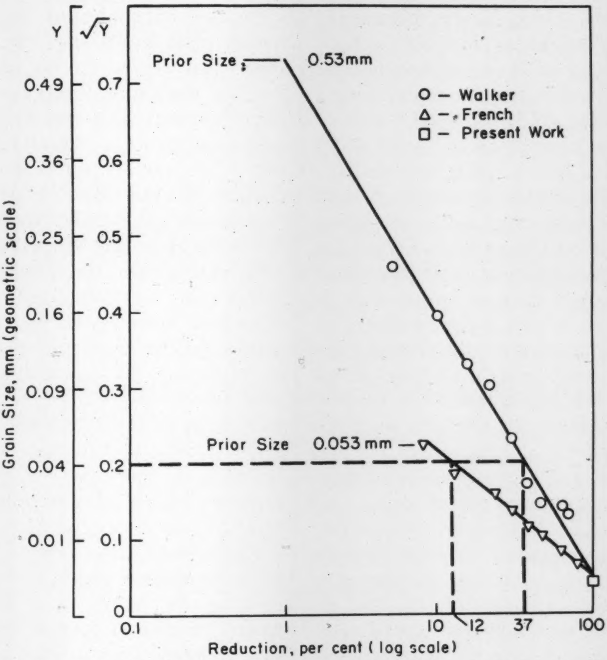


FIG. 5.—Conditions for Complete Recrystallization to Minimum Grain Size for 70-30 Brass.

The third feature of Fig. 3 is the fan-shaped array of the data. This array is important in defining the grain growth law. This particular arrangement is similar to that previously shown for aluminum but which has not been consistently evident in brass. In particular, Beck (4) shows data in a different arrangement. The reason for this difference is not known at this time.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] A minimum initial recrystallization temperature of 204 C has been reported for wire by Madigan and Blank (3).

The data in Fig. 3 are plotted on log-log coordinates because this choice most closely approximates linear fitting. A similar linear relationship is obtained when log grain size values are plotted as a function of the temperature squared in degrees Centigrade. These data are presented again in Fig. 4 to show time and temperature as the ordinates. Figure 4 is particularly important, for it is the base of reference for all the annealing results. Figure 4 will, therefore, be recognized in the basal plane of Figs. 1 and 2.

Above the basal plane in Figs. 1 and 2 are superimposed the grain growth data based on the results of Walker (5) and French (6). These data have been freely extrapolated on the basis that the only restriction to continued grain growth at higher temperature and longer time is the influence of the strip surfaces. In order to understand the interrelation between the new results and published work, Fig. 5 is introduced.

Figure 5 shows for the first time the combined effects of prior grain size and cold reduction on the minimum grain size for complete recrystallization, independent of annealing parameters. Walker (5) presented this information earlier but plotted log grain size as a function of square root of per cent reduction. The new selection of ordinates makes possible a more lucid interpretation of the effect of prior grain size. The square root of grain size is used, since grain growth is a surface energy effect and follows the geometric relationship between grain surface and volume. There appears to be no difference in the linear fit of the data as a result of this change of ordinates.

The impact of Fig. 5 is to make possible a more simplified statement of the various grain size effects in 70-30 brass. The question of preferred orientation, mixed structures, and origin of nuclei in rolling can be understood from a consideration of this figure. A description of the growth process may be valuable as a background for later remarks on these effects, as given in the following section.

GRAIN GROWTH PROCESS

Effect of Prior Grain Size:

In the annealing of brass from a coarse prior grain size, the regions of subcritical strain do not recrystallize. Instead, their strain energy is relieved by the different mechanisms involved in the relaxation process. Simultaneously, the new grain population is growing from centers of high strain to consume the contiguous less highly strained regions. When the annealing temperature is raised sufficiently high, the grain boundaries surrounding strain-free remnants of the prior grain structure become increasingly active and the larger prior grains proceed to cannibalize the new grain population. The process is for the grain boundaries to migrate so as to decrease the smallest grain surfaces and increase the adjacent larger ones. The only change in an elemental volume of the brass structure after a grain-boundary wave has passed is the change in its orientation to that of the new, more stable grain. This process may be quite rapid in structures where the disparity between prior grain size and new growth size is large. The process is rapid because of the relatively high energy state of the small grain areas and the favorable grain boundary angles between large and small grains. Increasing cold reduction retards the process of grain growth since there is less aid or "catalysis" from the presence of large prior grain remnants. The growth process after the most severe reductions finally reverts to the slow process of competition between grains of equal size in a uniform population. The ideal competitive growth process occurs in annealing from the extreme reduction shown at the right side of Fig. 5, or from reductions approaching the limit of cold rolling.

One important feature of Fig. 5 is that the data extrapolate to a single value at about 0.002 mm at the limit of cold rolling. This observation explains the fact that the annealed grain size after severe reductions is independent of prior grain size, because the minimum grain size curves for all prior reductions terminate at this point.

The effect of decreased prior grain size, for a constant cold reduction, is to lower the minimum grain size limit upon annealing. Figure 5 shows that for a minimum grain size of 0.040 mm, a 12 per cent reduction is required from an 0.053-mm prior grain size, whereas 37 per cent is required for an 0.53-mm prior grain size. With greater reductions, the disparity due to prior grain size becomes less and finally disappears.

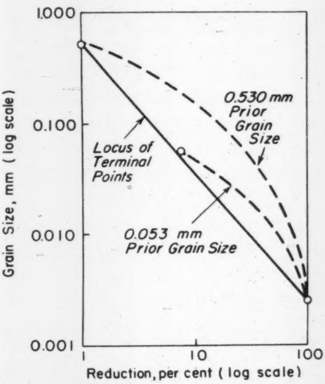


Fig. 6.—Logarithmic Relation Between Prior Grain Size and Minimum Reduction for Grain Refinement upon Annealing.

It will be observed on the left-hand side of Fig. 5 that the curves showing minimum grain size upon completion of recrystallization stop at a point where the final grain size and the prior grain size are the same. The reduction at which the prior grain size and the minimum annealed grain size are the same will be called the terminal reduction for that grain size. To the left of each terminal point, strains are relieved by recovery without recrystallization. To the right of the terminal point, recrystallization occurs at regions above critical strain simultaneously with recovery at regions below critical strain. The three terminal points shown (1 per cent, 9 per cent, and 99 per cent reduction) are three representatives of a family of terminal points whose locus is a concave downward curve not shown in this figure. This locus is shown as a straight line in Fig. 6, when the data are plotted in log-log coordinates. The lines for minimum grain size for each prior grain size lie in curved arcs above, as shown. The data for these arcs form the curved surfaces of complete recrystallization shown in Figs. 1 and 2.

Origin of Nuclei:

Further use can be made of Fig. 6 in regard to the initiation of nuclei during rolling. The terminal reduction for any grain size represents the point at which the first centers of critical strain are formed during rolling. At the limit of cold rolling, the whole strip may be regarded as converted to regions of critical strain. For any reduction between these limits, the fraction of the strip converted to regions of critical strain is in direct proportion to its position between these two limits.

The limiting minimum grain size indicated in Fig. 5 is artificial in that it is useful principally in a description of the grain growth process and refers specifically to the conditions of complete recrystallization in cold-rolled strip. It is not to be inferred that grains smaller than 0.002 mm are not observed. These data show that this is about the limiting size to be expected in fully recrystallized brass strip. Such grains are on the order of 10,000 atom diameters in size, are small compared to the blocky units to be found in the microstructure of severely rolled brass, and are large compared to what is imagined to be the size of the nucleus. It is unnecessary to make assumptions regarding the mechanisms of nucleation for the purpose of this paper.

The gap between the locus of terminal points and the minimum recrystallized grain size curve in Fig. 6 has another important significance. The gap measured on a vertical line is a measure of the increase in grain size contributed by the remnants of prior grains over the minimum grain size at the rolling limit (0.002 mm). The gap represents the maximum degree of "abnormality" that may occur in strip when insufficient cold work is used between a high-temperature prior anneal and a low-temperature final anneal.

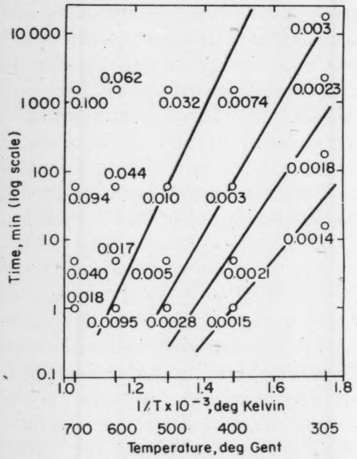


FIG. 7.—Activation Energy for Grain Growth in Fine Grained 70-30 Brass.

Activation Energy for Grain Growth:

The new data on grain growth after severe reductions is of interest in connection with the activation energy of grain growth. Figure 7 shows the data from Fig. 3 in the form sometimes used to facilitate activation energy calculations. Although the data in Figs. 3 and 4 do not conform to the reciprocal absolute temperature law, it can be argued that this law holds approximately and is valid for short intervals. On this basis, Fig. 7 shows an activation energy of 28.3 kg-cal at 0.0015 mm, with continuously increasing values at larger grain sizes and annealing temperatures. A re-plot of Walker's data (5) shows a similar array with higher activation energies to correspond with his larger grain size values.

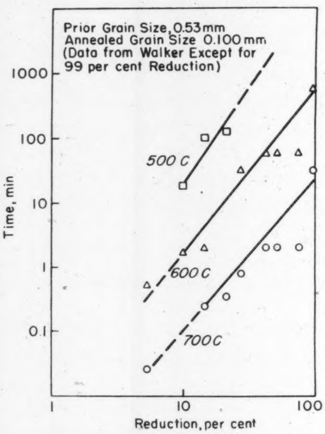


FIG. 8.—Effect of Cold Reduction on Annealing Time for Constant Annealed Grain Size.

The conclusion is that a single activation energy for grain growth in 70-30 brass does not exist. The initial activation energy is as low as 28.3 kg-cal, and perhaps may be found somewhat lower where smaller grain sizes are involved. The activation energy for grain growth, then, increases continuously to the melting point. Further, the activation energy for initial grain growth is substantially lower than that for diffusion, given in the literature near 39 kg-cal.

Control of Final Grain Size:

The importance of cold rolling to decrease the final grain size under constant annealing conditions is emphasized in Fig. 8. This figure is of considerable importance in two popular commercial annealing practices. First, the "flash" annealing or short-time strip annealing processes are practical because of the moderate grain sizes obtained with short-time, high-temperature exposure of more severely rolled strip. Second, the bell furnaces often used for long-time anneals and accurate temperature control are well suited to production of fine grain sized strip with freedom from "orange-peel" surface and with extraordinary mechanical properties. Grain size control for both these processes is practical without unusually accurate control of annealing time and temperature. Figure 8 is only one of a family of similar charts that can be derived from Figs. 1 and 2 for other final annealed grain sizes.

Preferred Orientation:

Although the grain size behavior of brass can be described in terms of the four primary variables, the use of prior grain size as a primary variable introduces a technicality of definition. It is theoretically possible for the grain structure at any time to consist of some grains which have survived a number of rolling and annealing cycles. As far as grain growth is concerned, however, the criterion of behavior is in the average grain size of the prior grain population, not in its origin. The grain origin has a different significance in the consideration of preferred orientation, for the grains which have survived successive processing cycles are oriented preferentially. It is interesting that such oriented fractions of the population apparently have little effect on grain growth rate in 70-30 brass.

Although these orientations may have little effect on grain growth, the reverse is certainly not true, as will be shown in the last section below. It is concluded that the thread of continuity among the recrystallization textures strongly depends upon the interplay between prior and final grain size.

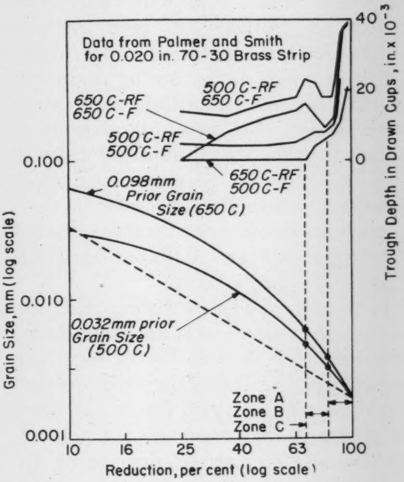


FIG. 9.—Effect of Prior Grain Size on Preferred Orientation.

The degree of orientation, or earing tendency, in annealed brass cannot be understood without some further knowledge of the prior history in addition to the four primary variables. Figure 9 shows some data from Palmer and Smith (7) re-plotted to correspond with the above figures where log per cent reduction was used as the ordinate. The appearance of the data plotted in this way is a distortion of Palmer and Smith's Fig. 8, but the purpose of these data is to bring out the relationship to the prior grain size in the manner developed in Fig. 6 above. This figure is selected from several of Palmer and Smith's which show the same important trends. They do not give the ready-to-finish grain size values, so an estimate was made of the values from their annealing temperatures and Figs. 1 and 2 above. The corresponding minimum grain sizes curves were drawn in Fig. 9, marked 0.098 mm and 0.032 mm.

These data show the strong double peak in the earing tendency with increasingly severe cold reductions. The method of plotting has been used to emphasize the preferred orientation, which in reality is not strong compared to that obtained in copper and some other alloys. No new information is gained by this procedure other than to demark more clearly the critical cold-reduction values. The range of reductions is cut into three zones by these critical reductions and are indicated as Zones *A*, *B*, and *C* in Fig. 9.

It is fairly well established that preferred orientation would not be observed in annealed strip rolled with less than about 75 per cent reduction from the hot-rolled or random grain structure. Brick (8) observed that about 90 per cent reduction was required to develop an appreciable rolling texture from the as-cast structure. He noted that the annealing texture intensity followed that of the rolling texture, increasing with increasing severity of rolling. The annealing texture he reports is the (113) [211] texture. Later Brick (9) observed the beginning of this annealing texture after somewhat more than 75 per cent reduction from a hot-rolled structure. Both of these were free from prior texture, confirming the true root annealing texture and the extent of zone *A*. Early in this zone, Burghoff and Bohlen (10) show the earing tendency is toward six ears, whereas after more severe reductions, four ears form. The six ears have been described from the appearance of their pole figures as a combination of the (110) [115] and (110) [011] orientations, occurring at 85 per cent reduction. The four ears appear to be best described as the (110) [112] orientation after 96 per cent reduction. These materials were not free of prior texture. The zone *A* orientation effects are complicated due to the interplay of residual textures, twinning, root texture, and perhaps the random tendency.

The question of the second peak annealing texture in 70-30 brass, indicated as zone *C* in Fig. 9, is fairly simple to understand. The coexistence of prior and final grain populations has been explained quantitatively in connection with Fig. 6. Even though the prior grain population has been entirely relieved of deformation stresses during the anneal, the rolling has imposed its orientating effect on the prior grains. This orientating effect is cumulative as long as the prior grains survive. Figure 9 shows that the low-temperature ready-to-finish anneal has produced a consistently higher earing tendency, due to the assured survival of large prior grain remnants whose preferred orientation improves with the accumulated effect of two rolling sequences. In the texture zone *C*, the orientation to survive is the (110) [112] rolling texture, for it is obvious that only the rolling texture can be accumulated. The rolling texture in zone *C* becomes more dominant with increasing reductions and final annealing temperatures (Fig. 9), as would be expected, until the zone *B* reductions are reached.

The zone *B* reductions range from about 69 per cent reduction to about 75 per cent. These reductions are sufficient to reduce the effective prior grain size to the range of about 0.006 to 0.003 mm as shown in Fig. 9. In these surroundings it is evident that the new grain population competes more favorably than it would with larger prior grains. In this way, the over-riding influence of the prior texture is reduced or eliminated.

SUMMARY

As a result of this work, certain areas of incomplete knowledge appear. For example, grain growth restricted to two dimensions has not been studied in brass. Also, the origin of annealing textures in zone *A* in brass is not entirely clear. These problems involve some experimental difficulties which are more involved than those encountered in the preceding investigations in this field and should yield interesting results concerning the mechanisms involved.

Acknowledgment:

The author appreciates very much the helpful discussions with W. Rostoker, Assistant Manager of Metals Research Department of Armour Research Foundation of Illinois Institute of Technology.

REFERENCES

(1) Discussion of Beck, Towers, and Manly paper (reference (4)).
(2) B. L. Averbach, "Recovery and Recrystallization in Brass," *Transactions*, Am. Inst. Mining Engrs., Vol. 185, p. 491 (1949).
(3) S. E. Madigan and A. I. Blank, "Recovery and Recrystallization in Long Time Annealing of 70-30 Brass," *Transactions*, Am. Inst. Mining Engrs., Vol. 137, p. 170 (1940).
(4) P. A. Beck, J. Towers, and W. D. Manly, "Grain Growth in 70-30 Brass," *Transactions*, Am Inst. Mining Engrs., Vol. 175, p. 162 (1948).
(5) H. L. Walker, "Grain Size Produced by Recrystallization and Coalescence in Cold Rolled Cartridge Brass," *University of Illinois Bulletin*, Vol. 43, No. 21, Nov. 23, 1945.
(6) R. S. French, "Grain Growth in 70-30 Brass," *Transactions*, Am. Inst. Mining Engrs., Vol. 156, p. 195 (1944).
(7) E. W. Palmer and C. S. Smith, "Effect of Some Mill Variables on the Earing of Brass in Deep Drawing," *Transactions*, Am. Inst. Mining Engrs., Vol. 147, p. 164 (1942).
(8) R. M. Brick, "Correlation of Deformation and Recrystallization Textures of Rolled 70-30 Brass," *Transactions*, Am. Inst. Mining Engrs., Vol. 137, p. 193 (1940).
(9) F. H. Wilson and R. M. Brick, "Textures, Anisotropy and Earing Behavior of Brass," *Transactions*, Am. Inst. Mining Engrs., Vol. 161, p. 173 (1945).
(10) H. L. Burghoff and E. C. Bohlen, "Directional Properties of 68-32 Brass Strip," *Transactions*, Am. Inst. Mining Engrs., Vol. 147, p. 144 (1942).

THE CREEP AND RUPTURE PROPERTIES OF THREE CAST COPPER-BASE ALLOYS BETWEEN 550 AND 650 F (290 AND 345 C)*

By H. V. Kinsey[1]

Synopsis

The creep and rupture properties of a nickel bronze Ni-Vee A, a leaded-nickel bronze Ni-Vee C and a copper-base casting alloy "Navy M," have been measured over the temperature range of 550 to 650 F (290 to 345 C). The effect of an aging treatment on the creep and rupture properties of the nickel bronze has also been determined. Due to excessive scatter of results it has only been possible to observe certain trends. The nickel bronze has better creep and rupture properties than either of the other two alloys tested at these temperatures. The heat treatment used for this nickel bronze causes a deterioration of its rupture properties.

One of the more recent additions to the bronze family has been a group of nickel-bearing bronzes identified by the trade name of "Ni-Vee." As part of a program in the Mines Branch Physical Metallurgy Division of The Canadian Department of Mines and Technical Surveys, to study the foundry characteristics and properties of several bronzes (**1**,**2**,**3**),[2] the elevated temperature creep and rupture properties of two of these Ni-Vee bronzes, and a nickel-free bronze, Navy "M," have been determined at 550, 600, and 650 F (290, 315, and 345 C). The effect of a solution and aging type of heat treatment on the elevated temperature creep and rupture properties of the lead-free Ni-Vee alloy has also been evaluated.

Experimental

The specified composition limits for two of the alloys tested and the nominal composition of the third are given in Table I. One of the Ni-Vee alloys tested, identified in this paper as "Ni-Vee," corresponds to the lead-free Ni-Vee A alloy. The other Ni-Vee alloy, identified in this paper as "Alloy No. 11," corresponds to the lead-bearing Ni-Vee C alloy.

All three alloys were tested in the "as-cast" condition. The Ni-Vee (Ni-Vee A) alloy was also tested in creep and stress-rupture in the solution-treated and aged condition. This heat treatment consisted of a solution treatment of 5 hr at 1400 F (760 C), oil quenched, and then an aging treatment of one week at 550 F (290 C).

The casting temperature of the metal was within the range of 2150 to 2030 F (1175 to 1110 C). A Dow-type test bar mold, similar to that shown in Fig. 6, was used to produce cast-to-shape test bars. This type of mold was used in order to get the maximum number of test bars from each melt of metal.

---

* Published by permission of the Director, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Ont. (Canada).

[1] Senior Scientific Officer, Physical Metallurgy Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Ont. (Canada).

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The actual chemical composition of the melts used in this work is given in Table II. Room temperature tension tests, obtained on unmachined test bars, are listed in Table III. Room temperature tensile values for melt No. 198 are not available. However, since this melt accounts for less than 10 per cent of the creep and rupture data reported for the Ni-Vee alloys and since the actual creep and rupture values obtained on melt No. 198 are consistent with those of the other Ni-Vee melts, it is not felt that this omission is serious.

TABLE I.—SPECIFIED CHEMICAL COMPOSITION OF ALLOYS TESTED.

Alloy Designation	Specification Source	Copper	Composition, per cent					
			Tin	Lead	Zinc	Nickel	Iron	Phosphorus
Ni-Vee (Ni-Vee A)	ASTM Specification B 292 – 56[3] for Alloy A	85.0 to 90.0	4.5 to 6.0	0.10 maximum	2.5 maximum	4.5 to 6.0	0.25 maximum	...
Alloy No. 11 (Ni-Vee C)	Nominal Composition	80	5	5	5	5	...	...
Navy M	ASTM Specification B 61 – 52[4]	86.0 to 90.0	5.5 to 6.5	1.0 to 2.0	3.0 to 5.0	1.0 maximum	0.25 maximum	0.05 maximum

TABLE II.—CHEMICAL COMPOSITION OF ALLOY MELTS TESTED.

Alloy	Melt	Composition, per cent						
		Copper	Nickel	Lead	Tin	Zinc	Iron	Phosphorus
Ni-Vee	No. 136	87.80	4.90	0.02	4.84	2.16	0.03	0.010
	No. 196	87.15	4.73	trace	5.26	2.76	0.03	0.005
	No. 198	87.90	4.80	trace	4.88	2.38	0.03	0.008
	No. 200	87.90	4.75	0.03	5.08	2.12	0.02	0.005
	No. 271	86.10	4.89	0.07	5.76	3.08	0.04	0.006
	No. 216	87.44	4.89	...	5.20	2.36	...	...
Alloy No. 11	No. 197	81.40	4.80	4.51	5.50	3.69	0.04	0.006
	No. 201	81.45	4.78	4.65	5.67	3.62	0.03	0.006
	No. 204	82.55	4.81	4.45	4.69	3.57	0.03	0.005
Navy M	No. 199	89.81	nil	1.44	5.70	3.10	0.02	0.007
	No. 205	90.50	nil	1.20	5.61	3.06	0.02	0.005

Elevated temperature tensile properties are available for some of the melts used in the creep and rupture testing program. These data are included in this paper in order to present as complete a set of mechanical properties as possible for these alloys. Tables IV, V, and VI list the tensile properties obtained at 550, 600, and 650 F (290, 315, and 345 C).

Creep and stress-rupture tests were made at 550 F (290 C), 600 F (315 C), 650 F (345 C), and 700 F (370 C). An

---

[3] Specification for Nickel-Tin Bronze Castings (B 292 – 56), 1958 Book of ASTM Standards, Part 2, p. 496.

[4] Specification for Steam or Valve Bronze Castings (B 61 – 52), 1958 Book of ASTM Standards, Part 2, p. 116.

TABLE III.—TENSILE PROPERTIES AT ROOM TEMPERATURE FOR AS-CAST ALLOYS.

Alloy Designation	Melt	Ultimate Tensile Strength, psi	Yield Strength, 0.2 per cent offset, psi	Elongation in 2 in., per cent
Ni-Vee (Ni-vee A)	ASTM Specification B 292 – 56	45 000 minimum	20 000 minimum	25 minimum
	No. 136[a]	44 600	19 450	29
	No. 196	50 700	20 700	33
	No. 200	50 250	21 425	36.5
	No. 271	48 100	23 100	27
	No. 216	46 600	21 200	38.5
Alloy No. 11 (Ni-Vee C)	No. 197	42 450	22 400	12
	No. 201	41 400	23 300	8.5
	No. 204	41 500	18 900	12
Navy M	ASTM Specification B 61 – 52	34 000 minimum	16 000 minimum	22 minimum
	No. 199	44 675	19 050	44
	No. 205	44 400	19 100	42

[a] No. specified minimum properties available from ASTM.

TABLE IV.—TENSILE PROPERTIES AT 550 F (290 C) FOR AS-CAST ALLOYS.

Alloy Designation	Melt	Ultimate Tensile Strength, psi	Yield Strength, 0.2 per cent offset, psi	Elongation in 2 in., per cent
Ni-Vee (Ni-Vee A)	No. 196	43 200	17 300	32
	No. 200	41 600	18 200	28
Alloy No. 22 (Ni-Vee C)	No. 197	33 750	17 800	10
	No. 201	34 400	17 400	8
Navy M	No. 199	30 800	14 500	19.8

TABLE V.—TENSILE PROPERTIES AT 600 F (315 C) FOR AS-CAST ALLOYS.

Alloy Designation	Melt	Ultimate Tensile Strength, psi	Yield Strength, 0.2 per cent offset, psi	Elongation in 2 in., per cent
Ni-Vee (Ni-Vee A)	No. 196	43 350	19 700	25
	No. 200	43 700	not available	27.8
Alloy No. 11 (Ni-Vee C)	No. 197	34 350	17 450	10.2
	No. 201	34 000	17 300	10.0
Navy M	No. 199	26 700	14 600	13

TABLE VI.—TENSILE PROPERTIES AT 650 F (345 C) FOR AS-CAST ALLOYS.

Alloy Designation	Melt	Ultimate Tensile Strength, psi	Yield Strength, 0.2 per cent offset, psi	Elongation in 2 in., per cent
Ni-Vee (Ni-Vee A)	No. 196	40 550	20 150	19
	No. 200	42 000	24 800	12.5
Alloy No. 11 (Ni-Vee C)	No. 197	29 350	17 900	8.0
	No. 201	28 000	17 300	6.0
Navy M	No. 199	16 800	14 200	3.1

attempt was made to design the testing program to give stresses for 0.05 and 0.1 per cent creep strain, 0.2 and 0.5 per cent total strain, and rupture in times up to 5000 hr at temperatures between 550 F (290 C) and 650 F (345 C). For the sake of brevity, the time *versus* strain curves for the tests conducted are not reproduced in this report. The data obtained from these curves are summarized in Tables VII, VIII, IX, and X.

DISCUSSION OF RESULTS

An examination of Tables VII to X will indicate that, particularly in the

TABLE VII.—SUMMARY OF CREEP AND RUPTURE TESTS OF AS-CAST Ni-VEE ALLOY.

Test Bar	Stress, psi	Time, hr					After Rupture	
		0.05 per cent Creep Strain	0.10 per cent Creep Strain	0.2 per cent Total Strain	0.5 per cent Total Strain	Rupture	Elongation in 2 in., per cent	Reduction of Area, per cent
TEST TEMPERATURE, 550 F (290 C)								
No. 271-A2	35 000	...	...	...	...	80	...	...
No. 271-A1	32 000	...	...	...	...	135	nil	nil
No. 271-E4	30 000	...	...	...	...	137	...	...
No. 198-E2	30 000	3	...	...	...	31	6.0	5.8
No. 271-M1	27 500	165	460	55	...	1056	nil	nil
No. 136-M1	27 500	167	650	...	...	1744	...	...
No. 216-F1	27 500	30	225	...	...	204	9.0	8.9
No. 198-A2	25 000	130	430	10	...	1211	nil	nil
No. 196-E2	20 000	100	400	120	2330	4428	...	...
TEST TEMPERATURE, 600 F (315 C)								
No. 271-E1	27 500	55	...	...	...	134	...	...
No. 200-F2	25 000	85	175	...	...	264	nil	nil
No. 271-E2	25 000	65	125	10	...	335	...	...
No. 136-L4	22 500	175	345	140	...	663	...	...
No. 216-A4	22 500	80	150	...	...	265	5.0	4.5
No. 271-M2	22 500	...	...	...	...	137	...	...
No. 200-K2	20 000	112	240	340	1310	1530	nil	nil
No. 196-C2	15 000	130	260	275	...	2136	...	...
No. 216-D3	12 500	295	575	840	...			
TEST TEMPERATURE, 650 F (345 C)								
No. 271-A3	22 500	...	...	10	...	79	...	...
No. 271-M3	20 000	...	47	27	...	526	...	...
No. 200-G2	20 000	40	76	15	...	162	nil	nil
No. 271-A4	19 000	30	68	25	...	312	...	...
No. 196-B2	13 000	60	135	140	1320	1461	...	...
No. 200-C2	12 000	80	220	275	...	2766	1.0	nil
No. 196-F2	10 000	80	170	260	4020	4724	nil	nil

TABLE VIII.—SUMMARY OF CREEP AND RUPTURE TESTS OF Ni-VEE ALLOY IN THE SOLUTION TREATED AND AGED CONDITION.

Test Bar	Stress, psi	Time, hr					After Rupture	
		0.05 per cent Creep Strain	0.1 per cent Creep Strain	0.2 per cent Total Strain	0.5 per cent Total Strain	Rupture	Elongation in 2 in., per cent	Reduction of Area, per cent
TEST TEMPERATURE 550 F (290 C)								
No. 196-G2	25 000	...	...	...	...	6	nil	nil
No. 196-J2	20 000	30	...	56	...	151		
No. 271-B1	20 000	...	...	...	...	189		
No. 271-D1	18 000	385	...	...	...	439		
No. 271-D2	17 000	...	...	...	...	406		
No. 198-C2	17 000	308	...	...	...	685		
No. 200-J2	15 000	600	2100	...	...	...	...	...
TEST TEMPERATURE, 600 F (315 C)								
No. 196-D2	20 000	...	...	...	...	7	nil	nil
No. 271-F1	17 500	...	...	...	...	31		
No. 198-D2	17 000	...	...	...	...	94		
No. 196-K2	15 000	...	...	...	...	53		
No. 200-D2	15 000	...	...	...	...	159		
No. 271-B2	15 000	...	...	...	...	315		
No. 271-F2	13 500	...	...	...	...	207		
No. 200-B2	12 500	...	...	...	...	103		
No. 200-A2	10 000	800	...	...	...	...	...	...
TEST TEMPERATURE, 650 F (345 C)								
No. 196-A2	15 000	...	...	...	...	154	nil	nil
No. 271-B3	15 000	...	...	...	...	31		
No. 198-B2	12 500	95	186	195	...	354		
No. 271-J1	12 500	...	...	...	...	79		
No. 200-B2	10 000	315	470	535	...	640		
No. 271-J2	9 000	245	410	460	665	677		

TABLE IX.—SUMMARY OF CREEP AND RUPTURE TESTS OF ALLOY NO. 11.

Test Bar	Stress, psi	Time, hr					After Rupture	
		0.05 per cent Creep Strain	0.10 per cent Creep Strain	0.20 per cent Total Strain	0.50 per cent Total Strain	Rupture	Elongation in 2 in., per cent	Reduction of Area, per cent
TEST TEMPERATURE, 550 F (290 C)								
No. 201-L2	25 000	...	...	...	...	113	2	2
No. 201-B2	22 500	...	...	...	...	375	3	1.6
No. 201-A2	20 000	20	145	...	600	1123	...	...
No. 197-E2	18 000	78	230	8	...	798	...	...
No. 201-C2	17 000	120	...	33	...	...	nil	nil
No. 201-H2	15 000	75	535	305	2680	2942		
No. 201-G2	14 500	75	160	25	...	...		
No. 204-B2	14 500	210	875	360	...	...		
TEST TEMPERATURE, 600 F (315 C)								
No. 204-A2	19 000	30	80	...	...	119	nil	nil
No. 204-F3	17 000	25	135	15	725	814		
No. 197-I2	15 000	30	100	70	...	798		
No. 197-J2	13 000	50	120	...	350	765		
No. 197-L2	11 500	45	200	277	1425	1501		
No. 204-A4	9 000	320	745	...	...	...	...	...
TEST TEMPERATURE, 650 F (345 C)								
No. 201-I2	15 500	...	...	...	...	22	nil	nil
No. 197-D2	13 000	8	20	20	...	241		
No. 197-A2	10 000	10	30	50	...	626		
No. 197-H2	8 500	15	65	110	...	293		
No. 201-F2	8 500	15	75	125	...	1293		
No. 201-J2	5 900	100	375	1220	...	...		

TABLE X.—SUMMARY OF CREEP AND RUPTURE TESTS OF ALLOY NAVY M.

Test Bar	Stress, psi	Time, hr					After Rupture	
		0.05 per cent Creep Strain	0.10 per cent Creep Strain	0.20 per cent Total Strain	0.50 per cent Total Strain	Rupture	Elongation in 2 in., per cent	Reduction of Area, per cent
TEST TEMPERATURE, 550 F (290 C)								
No. 199-I2	16 000	...	...	...	...	57	6	5.2
No. 199-L2	15 000	...	5	...	...	397	nil	nil
No. 199-E2	12 500	8	25	...	150	264	nil	nil
No. 205-B4	11 500	120	232	210	905	...	...	...
No. 205-A4	10 000	200	525	520	1725	...	...	...
TEST TEMPERATURE, 600 F (315 C)								
No. 199-G2	15 000	...	...	...	...	19	nil	nil
No. 199-F2	13 000	1½	3	...	...	38	4	5.3
No. 199-C2	11 000	15	50	20	352	599	nil	nil
No. 205-C4	10 000	25	65	80	250	...	...	...
TEST TEMPERATURE, 650 F (345 C)								
No. 199-H2	8 000	20	40	52	134	235	...	...
No. 199-J2	7 000	28	64	100	290	668	...	...
No. 199-D2	6 500	80	165	75	470	1094	3	3.1
TEST TEMPERATURE, 700 F (370 C)								
No. 205-E4	5 000	30	55	95	230	767	4	6.5

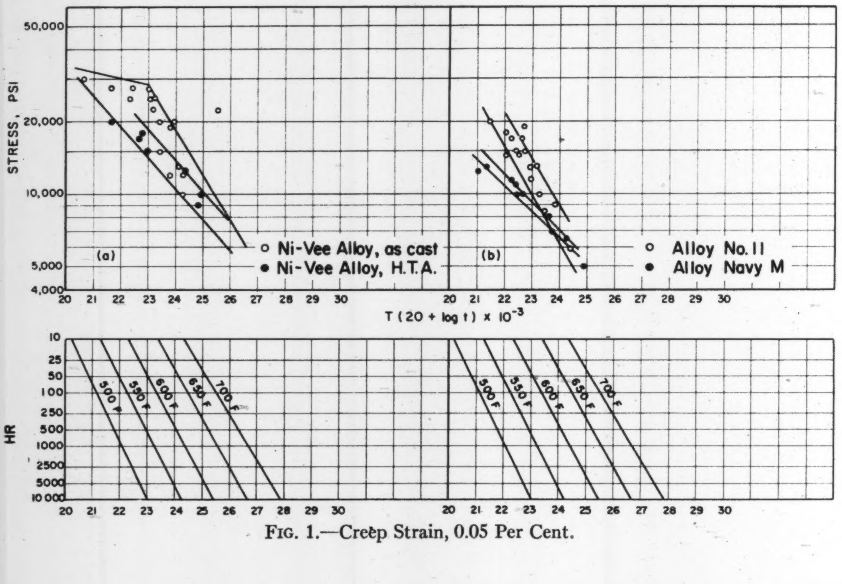


FIG. 1.—Creep Strain, 0.05 Per Cent.

case of the Ni-Vee alloy, there was a considerable amount of inconsistency in the results obtained. To obtain a common basis for comparison of these alloys, it was necessary to determine values for the stress to cause rupture, 0.10 per cent creep strain, etc., in some fixed time, such as 100 hr or 1000 hr, at the test temperature used. Plotting the data contained in Tables III to VI on the basis of log stress *versus* log time, which is a method commonly used for this purpose, was not fully satisfactory. It was found that the Larsen-Miller plot made it easier to handle the data.

In the Larsen-Miller plot, the stress is plotted against a parameter calculated by using the formula:

$$(T + 459)(20 + \log t)$$

where:

T = temperature, deg Fahr, and
t = time in hr.

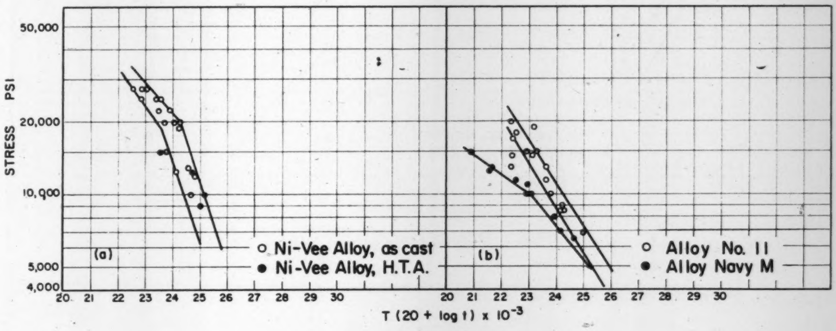


FIG. 2.—Creep Strain, 0.1 Per Cent.

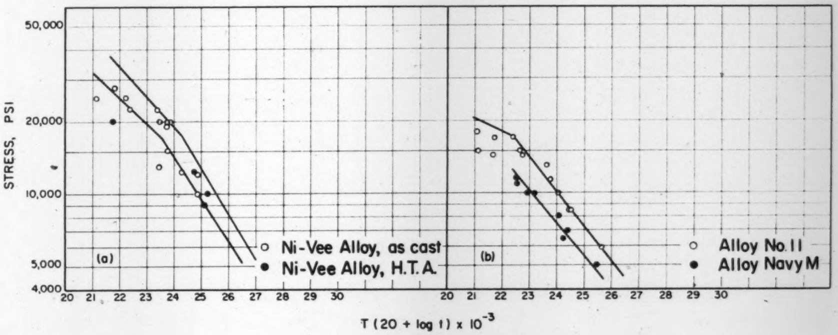


FIG. 3.—Total Strain, 0.2 Per Cent.

The plots are reproduced in Figs. 1 to 6. The 100-hr stresses and 1000-hr stresses obtained from these plots are listed in Tables XI to XIV. It is gratifying to note that the rupture stresses reported in Table X for the Navy M alloy at 550 F (290 C) are in good agreement with those recently reported by Simmons and Kura (**4**).

The most obvious conclusion to be drawn from an examination of these tables is that in order of decreasing stress rupture strength, these alloys may be listed as follows:

Ni-Vee alloy, as cast,
Alloy No. 11, as cast,
Ni-Vee alloy, solution treated and aged, and
Alloy Navy M.

From an examination of Figs. 1(*a*), 2(*a*), 3(*a*), and 4(*a*), it is apparent that the solution and aging treatment given Ni-Vee alloy did not affect its creep properties but did reduce its rupture strength. The data in Tables VIII and XII indicate that at 550 F (290 C) and 600 F (315 C) heat-treated Ni-Vee alloy ruptures before 0.1 per cent creep strain or 0.2 per cent total strain is reached.

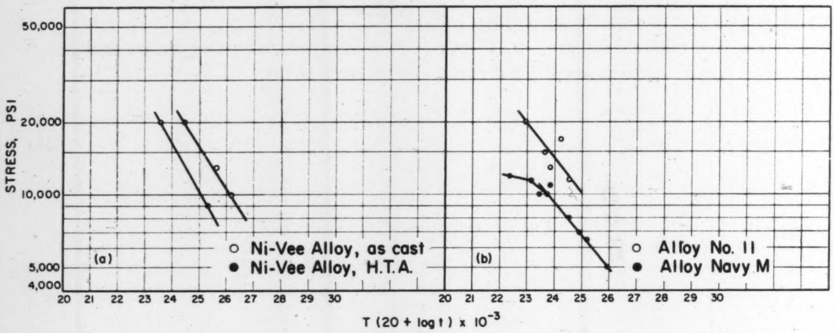


FIG. 4.—Total Strain, 0.5 Per Cent.

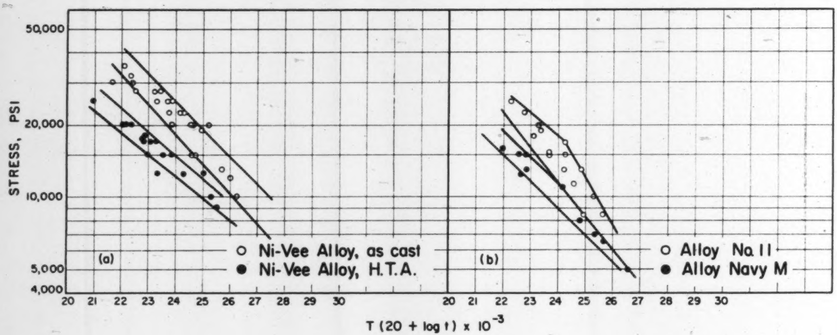


FIG. 5.—Rupture.

The addition of lead to the Ni-Vee composition, as represented by alloy No. 11, reduced the rupture strength by from 35 to 40 per cent over the temperature range tested.

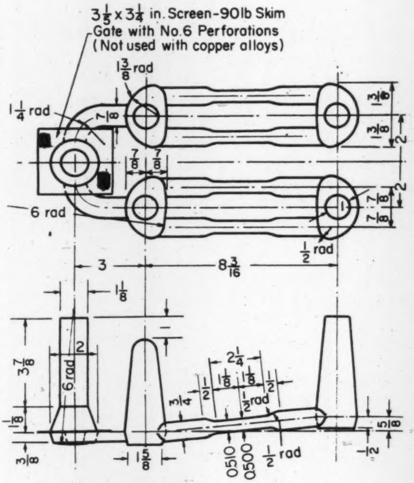
Alloy Navy M is weaker in all respects than any of the other alloys tested, on the basis of creep and rupture strengths up to 1000 hr duration.

CONCLUSIONS

The following conclusions are drawn from this work:

1. The alloys tested may be listed in order of decreasing stress rupture strength over the temperature range used, as follows:

Ni-Vee (Ni-Vee A) alloy, as cast,
Alloy No. 11 (Ni-Vee C), as cast,
Ni-Vee (Ni-Vee A) alloy, solution treated and aged, and
Alloy Navy M.


$3\frac{1}{5}$ x $3\frac{1}{4}$ in. Screen-90 lb Skim
Gate with No. 6 Perforations
(Not used with copper alloys)
$1\frac{3}{8}$ rad
$1\frac{1}{4}$ rad
$\frac{7}{8}$
$1\frac{3}{8}$
$1\frac{3}{8}$
2
$\frac{7}{8}$
$\frac{7}{8}$
2
1
$\frac{7}{8}$
$\frac{7}{8}$
6 rad
$\frac{1}{2}$ rad
3
$8\frac{3}{16}$
$1\frac{1}{8}$
1
2
$3\frac{7}{8}$
6 rad
$2\frac{1}{4}$
$\frac{1}{2}$
$1\frac{1}{8}$
$1\frac{1}{8}$
$\frac{1}{2}$
$\frac{3}{4}$
$\frac{1}{2}$ rad
$\frac{1}{8}$
$\frac{5}{8}$
$\frac{3}{8}$
$1\frac{5}{8}$
0.510
0.500
$\frac{1}{2}$ rad
$\frac{1}{2}$

Note: All dimensions in inches

FIG. 6.—Dow-Type Test Bar Mold to Produce Cast-to-Shape Test Bars.

2. The solution and aging treatment of the Ni-Vee (Ni-Vee A) alloy, as cast, does not affect its creep properties but only reduces its rupture strength.

3. The alloys tested may be listed in order of decreasing creep resistance at the test temperatures as follows:

Ni-Vee (Ni-Vee A) alloy, both conditions,
Alloy No. 11, (Ni-Vee C), and
Alloy Navy M.

4. It should be pointed out that, owing to the inconsistencies found among the test bars used in this program, it is not possible to do more than indicate probable trends in properties. There is a strong indication that the creep and rupture properties of these alloys are very sensitive to melting and casting conditions. Before a more exact study of the creep and rupture properties of these alloys is undertaken, it would be necessary to make a very exhaustive study of the influence of melting and casting variables on these properties.

TABLE XI.—100-HR AND 1000-HR CREEP AND RUPTURE STRESSES AT 550, 600, AND 650 F (290, 315, 345 C) FOR Ni-Vee ALLOY AS-CAST.

Property	550 F (290 C)		600 F (315 C)		650 F (345 C)	
	100-hr Stress, psi	1000-hr Stress, psi	100-hr Stress, psi	1000-hr Stress, psi	100-hr Stress, psi	1000-hr Stress, psi
0.05 per cent creep strain	17 000 to 30 000	13 000 to 26 000	12 000 to 23 000	9 000 to 15 000	9 000 to 15 000	...
0.10 per cent creep strain	30 000	22 000 to 28 000	21 000 to 27 000	10 000 to 18 000	9 000 to 16 000	4 000 to 7 000
0.20 per cent total strain	23 000 to 29 000	19 000 to 23 000	18 000 to 22 000	12 500 to 17 500	12 000 to 17 000	8 000 to 1 000
0.50 per cent total strain	...	...	...	13 000 to 20 000	...	...
Rupture	30 000 to 40 000	24 000 to 32 000	23 000 to 30 000	17 000 to 23 000	16 000 to 22 000	12 000 to 16 500

TABLE XII.—100-HR AND 1000-HR CREEP AND RUPTURE STRESSES AT 550, 600 AND 650 F (290, 315 AND 345 C) FOR Ni-Vee ALLOY SOLUTION TREATED AND AGED.

Property	550 F (290 C)		600 F (315 C)		650 F (345 C)	
	100-hr Stress, psi	1000-hr Stress, psi	100-hr Stress, psi	1000-hr Stress, psi	100-hr Stress, psi	1000-hr Stress, psi
0.05 per cent creep strain	17 000 to 23 000	13 000 to 17 000	12 000 to 16 000	9 000 to 12 000	9 000 to 12 000	...
0.10 per cent creep strain	...	...	...	...	9 000 to 16 000	4 000 to 7 000
0.20 per cent total strain	...	...	...	...	...	8 000 to 10 000
0.50 per cent total strain	...	...	...	...	...	...
Rupture	17 500 to 21 500	14 000 to 17 500	13 800 to 17 000	11 000 to 13 800	11 000 to 13 800	8 600 to 10 500

TABLE XIII.—100-HR AND 1000-HR CREEP AND RUPTURE STRESSES AT 550, 600 AND 650 F (290, 315 AND 345 C) FOR ALLOY NO. 11.

Property	550 F (290 C)		600 F (315 C)		650 F (345 C)	
	100-hr Stress, psi	1000-hr Stress, psi	100-hr Stress, psi	1000-hr Stress, psi	100-hr Stress, psi	1000-hr Stress, psi
0.05 per cent creep strain	14 000 to 20 000	9 400 to 13 000	8 600 to 12 000	5 500 to 7 800	5 500 to 7 800	...
0.10 per cent creep strain	18 000 to 20 000	12 500 to 16 000	12 000 to 15 000	7 500 to 9 500	7 500 to 9 500	4 500 to 6 000
0.20 per cent total strain	17 500	13 000	12 000	8 800	8 800	6 000
0.50 per cent total strain	...	18 500	17 500	13 000	13 000	...
Rupture	20 000 to 26 000	15 000 to 22 000	14 000 to 21 000	10 000 to 16 000	10 000 to 16 000	7 200 to 10 000

TABLE XIV.—100-HR AND 1000-HR CREEP AND RUPTURE STRESSES AT 550, 600 AND 650 F (290, 315 AND 345 C) FOR ALLOY NAVY M.

Property	550 F (290 C)		600 F (315 C)		650 F (345 C)	
	100-hr Stress, psi	1000-hr Stress, psi	100-hr Stress, psi	1000-hr Stress, psi	100-hr Stress, psi	1000-hr Stress, psi
0.05 per cent creep strain	10 500 to 12 000	8 000 to 9 400	7 800 to 8 800	6 000 to 6 800	6 000 to 6 800	...
0.10 per cent creep strain	12 000	9 000	8 800	6 500	6 500	4 700
0.20 per cent total strain	12 000	9 500	9 000	6 400	6 400	4 700
0.50 per cent total strain	12 500	11 500	11 000	8 000	8 000	5 500
Rupture	14 000 to 18 000	11 000 to 14 000	10 500 to 13 500	8 000 to 10 000	8 000 to 10 000	6 000 to 7 000

Acknowledgments:

The author acknowledges the help of Mr. J. O. Edwards, Head, Nonferrous Metals Section, and his staff, who supplied the material for this testing program, and of Mr. N. B. Brown, Head, Mechanical Testing Section, and his staff, for the tension testing involved.

REFERENCES

(1) J. O. Edwards, "The Effect of Prolonged Elevated Temperature Aging on the Room Temperature Properties of Several Cast Nickel Bronzes," *Research Report No. PM170,* Division of Physical Metallurgy, Mines Branch, Department of Mines and Technical Surveys, Ottawa (Canada), Aug. 20, 1954.

(2) J. O. Edwards, "The Effect of the Lead Content on the Room Temperature Mechanical Properties and Microstructure of Ni-Vee Alloy," *Research Report No. PM175,* Division of Physical Metallurgy, Mines Branch, Department of Mines and Technical Surveys, Ottawa (Canada), Dec. 1, 1954.

(3) J. O. Edwards, "Short-Time Elevated Temperature Properties of Three Copper-Base Casting Alloys," *Transactions,* American Foundrymen's Soc., Vol. 63, pp. 267–269 (1955).

(4) W. F. Simmons and J. G. Kura, "The Creep Properties of Three Low-Shrinkage, Copper-Base Casting Alloys," *Proceedings,* Am. Soc. Testing Mats., Vol. 58, p. 791 (1958).

DENSITY MEASUREMENTS ON STRAIN-CYCLED 2S ALUMINUM

By J. F. Tavernelli[1] and L. F. Coffin, Jr.[1]

Synopsis

The change in density was employed as a technique for the study of fatigue damage in 2S aluminum for various amplitudes and repetitions of fully reversed cyclic strain. Following diametral-strain amplitudes as large as 10 per cent for up to 80 per cent of the fatigue life, density measurements were made by a method sensitive to ±0.02 per cent. Density variations were found to be negligible.

As part of a study of the strain-cycling behavior of metals, an investigation was undertaken to explore the changes, if any, in the density of annealed 2S aluminum after various amounts of cyclic strain. Maier (**1**),[2] in his investigation of the density changes of cold-worked copper and Armco iron, found that there is a decrease of 1 per cent in density from the effect of cold work and that, after reaching a minimum value, the density again starts to increase (Fig. 1). Heindlhofer and Wright (**2**) showed that subsequent annealing only partially restores the density to its original value, as evidenced by their measurements of the change of density of steel during annealing. Thompson (**3**), in discussing the origin of fatigue cracks, has suggested that when both vacancies and interstitials remain near their plane of origin, a great concentration of them is produced so that the lattice is almost destroyed, a volume increase occurs, and the two parts of the crystal are forced apart. This would cause a decrease in density.

It was thought that with the introduction of cyclic plastic strain into a specimen the measurement of the density would give evidence of crystal imperfection, void formation, and other defects, which could be correlated with the amplitude and number of repetitions of cyclic strain. The present paper reports the results of this study.

Materials

The metal chosen for this investigation was commercially pure aluminum. Specimens of the form and dimensions shown in Fig. 2 were machined from wrought bar stock and annealed. They were then strain-cycled at various levels for predetermined numbers of cycles. A slug as shown in Fig. 3 was then cut from the central section of the strain-cycled specimen for use in the density measurements.

Testing Methods

The investigation was divided into two parts: strain-cycling and density measurement.

---

[1] Metallurgy and Ceramics Research Department, General Electric Research Laboratory, Schenectady, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The strain-cycling was conducted on apparatus previously described (4). Specimens were strain-cycled as shown in Fig. 4, always loading first in compression.

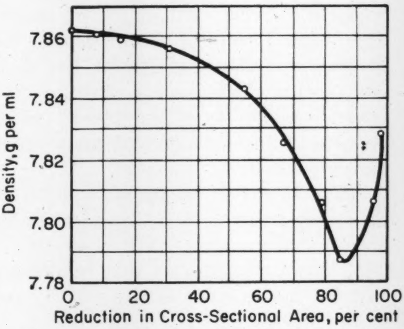


FIG. 1.—Effect of Cold Work (Drawing) on Density at 20 C of Armco Iron Wire, Annealed at 1026 C Prior to Drawing. (After Maier (1).)

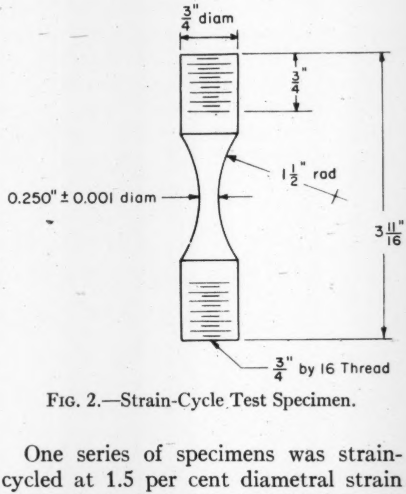


FIG. 2.—Strain-Cycle Test Specimen.

One series of specimens was strain-cycled at 1.5 per cent diametral strain (5) for ¼, ½, ¾, 1, 50, 100, 100¼, 100½, 100¾, 150, and 200 cycles. A second series of specimens was strain-cycled at 10 per cent diametral strain for ¼, ½, ¾, 1, 2, 3, 4, 4¼, 4½, and 4¾ cycles. Reference to Fig. 4 will show why these numbers of cycles were chosen. One-quarter-cycle specimens are subjected only to the compressive strain; one-half-cycle specimens to the compressive strain and returned to a zero strain condition; three-fourths-cycle specimens to the compressive and tensile strain; and one-cycle specimens to the compressive and tensile strain and returned to a zero strain condition.

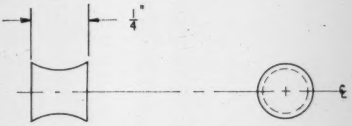


FIG. 3.—Slug from Strain-Cycle Test Specimen for Density Measurement.

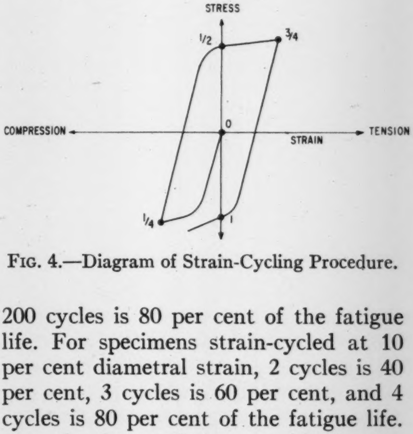


FIG. 4.—Diagram of Strain-Cycling Procedure.

For specimens strain-cycled at 1.5 per cent diametral strain, 100 cycles is 40 per cent, 150 cycles is 60 per cent, and 200 cycles is 80 per cent of the fatigue life. For specimens strain-cycled at 10 per cent diametral strain, 2 cycles is 40 per cent, 3 cycles is 60 per cent, and 4 cycles is 80 per cent of the fatigue life. Any density variations could thus be related to the developing fatigue process. An annealed specimen with no strain-cycling was used for a control specimen.

Density was measured by the flotation method (6), using tribromofluoromethane as a fluid with tetrahydrofurfuryl alcohol as an inhibitor against corrosion. A well-stirred bath and two thermometers for temperature measurements accurate to

0.1 C were employed. The average of two or three density measurements at 30.0 C was reported, and the accuracy of determination was between ±0.0003 and ±0.0006 g per ml.

TABLE I.—DENSITY MEASUREMENTS OF 2S ALUMINUM.

Specimen Number	Diametral Strain, per cent	Number of Cycles	Density, g per ml	Variation in Density, g per ml
1........	1.5	¼	2.7129	0.0003
2........	1.5	½	2.7140	0.0003
3........	1.5	¾	2.7137	0.0003
7........	1.5	100¼	2.7138	0.0003
8........	1.5	100½	2.7139	0.0003
9........	1.5	100¾	2.7135	0.0003
10........	10	¼	2.7134	0.0003
11........	10	½	2.7127	0.0003
12........	10	¾	2.7137	0.0003
13........	10	4¼	2.7132	0.0003
14........	10	4½	2.7141	0.0003
15........	10	4¾	2.7136	0.0003
D-1........	1.5	1	2.7139	0.0005
D-2........	1.5	50	2.7135	0.0005
D-3........	1.5	100	2.7139	0.0005
D-4........	1.5	150	2.7133	0.0005
D-5........	1.5	200	2.7132	0.0006
D-20R....	10	1	2.7126	0.0005
D-17......	10	2	2.7137	0.0006
D-18......	10	3	2.7130	0.0006
D-21......	10	4	2.7128	0.0006
Control...	none	none	2.7131	0.0005

TEST RESULTS AND DISCUSSION

Table I lists the specimen number, per cent strain, number of cycles, and the measured density. Figures 5 and 6 show density variations of 2S aluminum strain-cycled at 1.5 per cent diametral strain, while Figs. 7 and 8 show the density for 10 per cent diametral strain. The data are plotted with bands showing the limits of reliability of the density measurements (dotted lines) and the average values (solid lines).

There seems to be no evidence from this investigation that a measurable density change occurs with cyclic straining. The observed variation in density over the entire range of strain and number of cycles is very small—2.7126 to 2.7141, or 1 part in 2000. Although some variations do indeed occur, as is seen in Figs. 5 to 8, there appears to be no consistent and reasonable trend discernible. For example, in Figs. 5 and 7 differences in density are observed associated with the various portions of a single hysteresis loop for both 1.5 and 10 per cent diametral strain amplitudes for both the first cycle and after a sig-

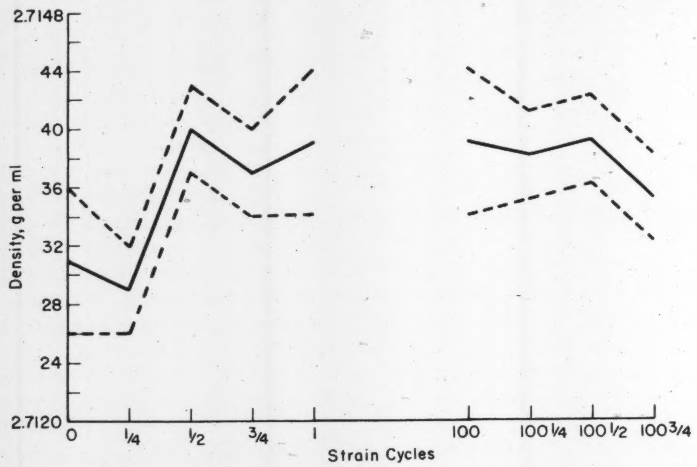


FIG. 5.—Density *Versus* Strain Cycles of 2S Aluminum Strain-Cycled at 1.5 per cent Diametral Strain.

nificant fraction of the total cyclic life. Close examination of these figures reveals, however, that the oscillations in density for 1.5 per cent amplitude are 180 deg out of phase with those for 10 per cent amplitude. A case might be made for a slight decrease in density for 1.5 per cent diametral strain with continued strain cycling (Fig. 6); however, the reality of this decrease is rendered

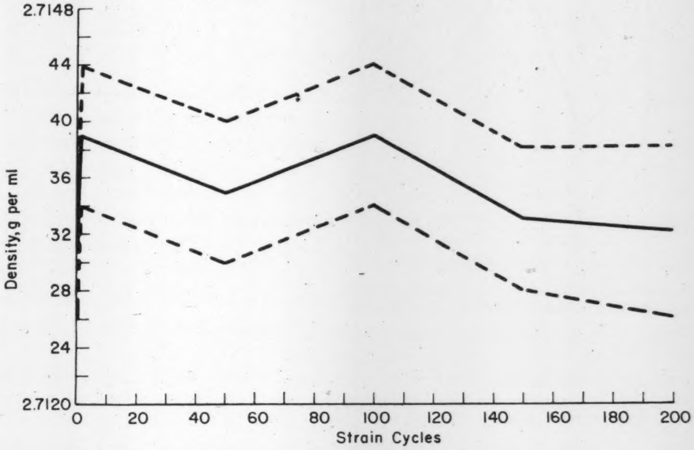


FIG. 6.—Density *Versus* Strain Cycles of 2S Aluminum Strain-Cycled at 1.5 per cent Diametral Strain.

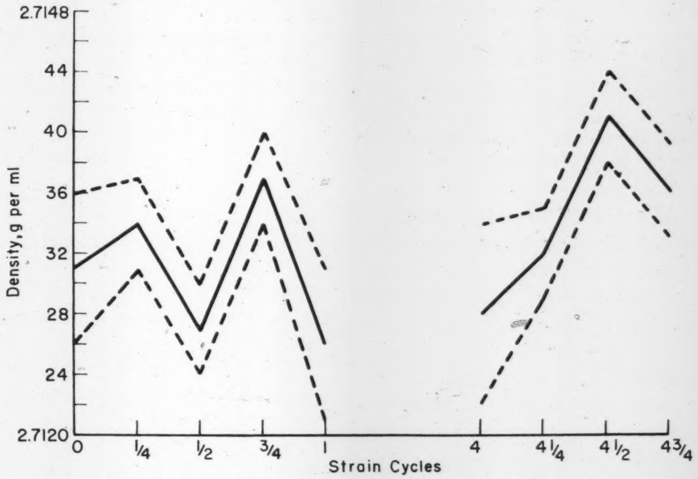


FIG. 7.—Density *Versus* Strain Cycles of 2S Aluminum Strain-Cycled at 10 per cent Diametral Strain.

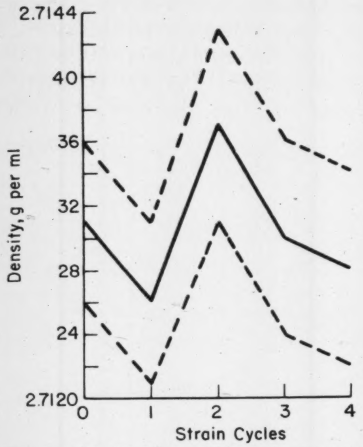


FIG. 8.—Density *Versus* Strain Cycles of 2S Aluminum Strain-Cycled at 10 per cent Diametral Strain.

doubtful by the uncertainty in the measurement. Furthermore, a diametral strain amplitude of 10 per cent (equivalent to a longitudinal strain range of 40 per cent) produces no significant density change either after a single loading or upon repeated loads.

CONCLUSION

Density variations were measured by a method sensitive to ±0.02 per cent and found to be negligible for 2S aluminum subjected to cyclic strains as large as 10 per cent diametral strain amplitude for up to 80 per cent of the fatigue life. Thus this method appears to be of little use as a means of studying the occurrence and development of fatigue damage in metals.

REFERENCES

(1) C. G. Maier, "Theory of Metallic Crystal Aggregates," *Transactions,* Am. Inst. Mining Engrs., Vol. 122, p. 121 (1936).

(2) K. Heindlhofer and F. L. Wright, "Density and X-ray Spectrum of Hardened Ball Steel Tempered at Various Temperatures," *Transactions,* Am. Soc. Steel Treating, Vol. 7, p. 34 (1925).

(3) N. Thompson, "Experiments on the Origin of Fatigue Cracks," in "Fatigue in Aircraft Structures," A. M. Freudenthal, editor, Academic Press, New York, 1956.

(4) J. F. Tavernelli and L. F. Coffin, Jr., "A Compilation and Interpretation of Cyclic-Strain Fatigue Tests on Metals," *Transactions,* Am. Soc. Metals, Vol. 51, p. 438 (1959).

(5) L. F. Coffin, Jr. and J. F. Tavernelli, "The Cyclic Straining and Fatigue of Metals," to be published in *Transactions,* Am. Inst. Mining and Metallurgical Engrs. (1959).

(6) E. L. Simons, J. S. Gutowski, and H. W. Middleton, Internal Memorandum, G. E. Research Laboratory, Schenectady, N. Y.

CREEP-RUPTURE BEHAVIOR OF NOTCHED AND UNNOTCHED SPECIMENS OF TYPES 304, 316, AND 321 AUSTENITIC STAINLESS STEELS*

F. GAROFALO[1]

SYNOPSIS

The creep-rupture behavior of types 304, 316, and 321 austenitic stainless steels was investigated at 1100 and 1500 F using unnotched and notched specimens. Based on the ratio of the notched creep-rupture strength to the unnotched creep-rupture strength for equivalent rupture times, types 304 and 321 are notch-strengthened for short rupture times at 1100 F. For intermediate rupture times a slight notch-weakening effect is found, but beyond about 1000 hr a tendency toward notch strengthening for both steels is again found. For type 316 material, a mild notch-strengthening effect is found at 1100 F for all periods studied. At 1500 F all three steels exhibit notch strengthening, type 321 showing the greatest degree of strengthening and type 304 the least.

An investigation of rupture initiation and propagation at 1100 F in notched specimens of type 321 indicates that rupture initiates just beneath the notch surface and propagates intergranularly toward the notch surface and to the interior simultaneously. An appreciable amount of plastic deformation follows after the propagating crack is apparently arrested, developing triaxial stresses at the specimen center and initiating a new crack which propagates outward and joins the arrested crack.

Most of the experimental results on creep and creep-rupture behavior of metals are determined on unnotched specimens under constant load and temperature. In service, however, components operating at elevated temperatures may be subjected to many variable conditions during normal or abnormal operation. For example, cycling temperatures and loads, nonsymmetric stress distributions, multiaxial stresses, and other factors are not uncommon. In recent years some effort has been made to determine the effect of many of these factors on creep and creep-rupture behavior in order that the adequacy of high-temperature alloys for various service conditions could be evaluated. In most of the studies that have been conducted, the principal interest has been to determine the effect of various factors on ductility and strength. Relatively little experimental work has been devoted to the study of fundamental aspects of flow and rupture behavior in engineering materials under various test conditions.

In the present paper, the effect of notches in tension bars on creep-rupture strength and rupture behavior is reported. A series of creep-rupture tests

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Edgar C. Bain Laboratory for Fundamental Research, U. S. Steel Corp., Research Center, Monroeville, Pa.

were made on unnotched and notched specimens of types 304, 316, and 321 austenitic stainless steels under various constant initial loads at 1100 and 1500 F. A detailed study of creep-rupture behavior in notched samples of type 321 material at 1100 F is also included.

TABLE I.—IDENTIFICATION AND CHEMICAL COMPOSITION OF STEELS TESTED.

AISI Type	Nominal Type	Chemical Composition, weight per cent										
		Carbon	Manganese	Phosphorus	Sulfur	Silicon	Nickel	Chromium	Molybdenum	Titanium	Nitrogen	Aluminum
304...	18-8	0.06	0.38	0.011	0.009	0.39	9.56	18.71	*x*	*x*	*x*	*x*
316...	18-8 Mo	0.05	1.73	0.026	0.015	0.57	13.09	17.52	2.25	*x*	0.026	*x*
321...	18-8 Ti	0.085	0.48	0.012	0.007	0.61	11.28	18.50	*x*	0.33	0.018	0.005

x—Not determined.

TABLE II.—HEAT TREATMENT, GRAIN SIZE, AND HARDNESS OF STEELS TESTED.

AISI Type	Heat Treatment			Grain Size	Hardness, Diamond Pyramid Number[a]
	Temperature, deg Fahr	Time, min	Quench		
304....	1950	60	Water	5 to 6	178
316....	2000	30	Water	5 to 6	151
321....	1950	30	Water	2 to 8	158

[a] Load = 10 kg.

MATERIALS AND TEST PROCEDURES

The chemical composition of the three austenitic stainless steels tested (types 304, 316, and 321) is given in Table I. Types 304 and 316 were received in the hot-rolled condition as 1-in. round bars. Type 321 was received in the forged condition as 1-in. diameter rounds. It may be noted for this material that the carbon is slightly higher and the titanium-carbon ratio lower than required by the AISI specification for type 321 steel.

As indicated in Table II, types 304 and type 321 were annealed by quenching from 1950 F; type 316 was quenched from 2000 F. Hardness and grain size of the annealed materials are also given in Table II.

Unnotched creep-rupture test specimens having a reduced section 0.252 in. in diameter and 1.25 in. in length were prepared and tested by previously established procedures (**1**.)[2] Notched creep-rupture specimens were prepared with one of the two notch geometries shown in Fig. 1. In types 304 and 316, a 45-deg notch was employed (Fig. 1(*a*)). The notch geometry used for these steel leads to a calculated stress concentration factor (ratio of maximum to average longitudinal stress, assuming elastic behavior) of about 3.7. For type 321 a 60-deg notch (Fig. 1(*b*)), was employed and the notch geometry, in particular the smaller radius of curvature at the base of the notch, leads to a higher stress concentration factor, the new factor being about 8.0. In the case of the type 321 material not all notched specimens tested were carried to complete rupture. Such partially failed specimens after various times under load were sectioned and prepared for metallographic examination to study the initiation and propagation of rupture.

CREEP-RUPTURE TEST RESULTS

The experimental results obtained for both unnotched and notched specimens tested at 1100 and 1500 F are shown in

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Figs. 2 and 3. Logarithmic plots of initial stress *versus* rupture time for the steels and test conditions employed are shown in Fig. 2(*a*, *b*, *c*). The 100 and 1000 hr rupture strengths are summarized in Table III.

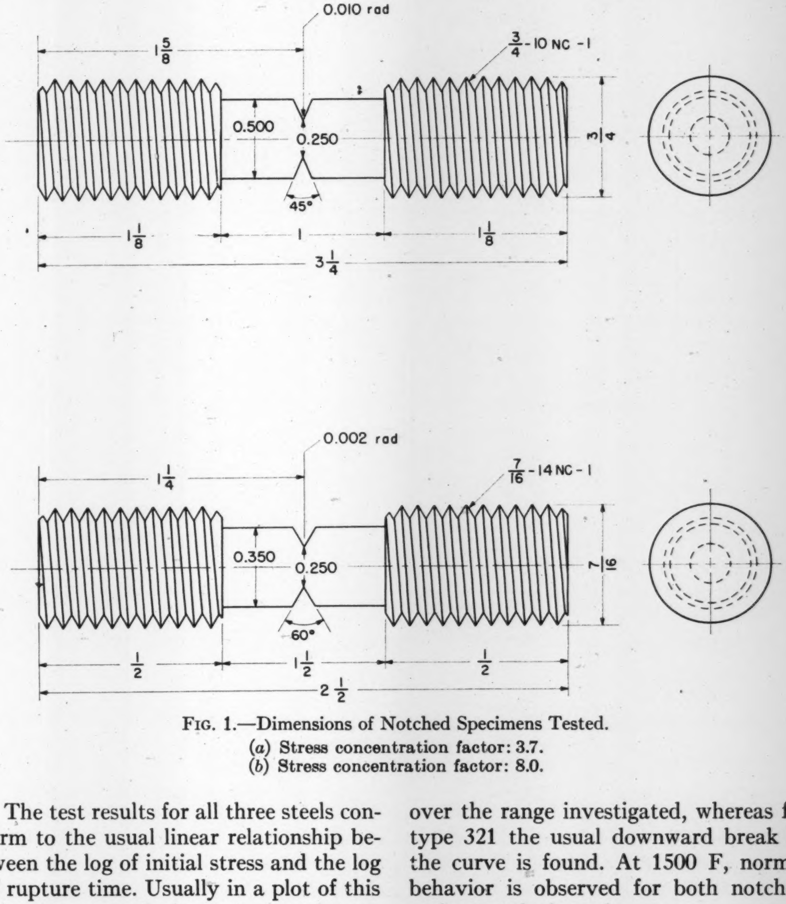

0.010 rad
$1\frac{5}{8}$
$\frac{3}{4}$-10 NC-1
0.500
0.250
$\frac{3}{4}$
45°
$1\frac{1}{8}$
1
$1\frac{1}{8}$
$3\frac{1}{4}$
0.002 rad
$1\frac{1}{4}$
$\frac{7}{16}$-14 NC-1
0.350
0.250
$\frac{7}{16}$
60°
$\frac{1}{2}$
$1\frac{1}{2}$
$\frac{1}{2}$
$2\frac{1}{2}$

FIG. 1.—Dimensions of Notched Specimens Tested.
(*a*) Stress concentration factor: 3.7.
(*b*) Stress concentration factor: 8.0.

The test results for all three steels conform to the usual linear relationship between the log of initial stress and the log of rupture time. Usually in a plot of this type for unnotched specimens, the experimental points lie on one or two linear segments, and when two linear segments are present the slope of the second segment (longer time) is greater than that of the first. It is therefore of interest to find that for the notched samples of all three steels tested at 1100 F the slope of the second linear segment is less than that of the first. For unnotched specimens tested at 1100 F, the data for types 304 and 316 plot as single straight lines over the range investigated, whereas for type 321 the usual downward break in the curve is found. At 1500 F, normal behavior is observed for both notched and unnotched specimens.

The reason for the inflection, or more generally termed instability, commonly found in the log stress - log rupture time relationship, is not understood and is being investigated at present.

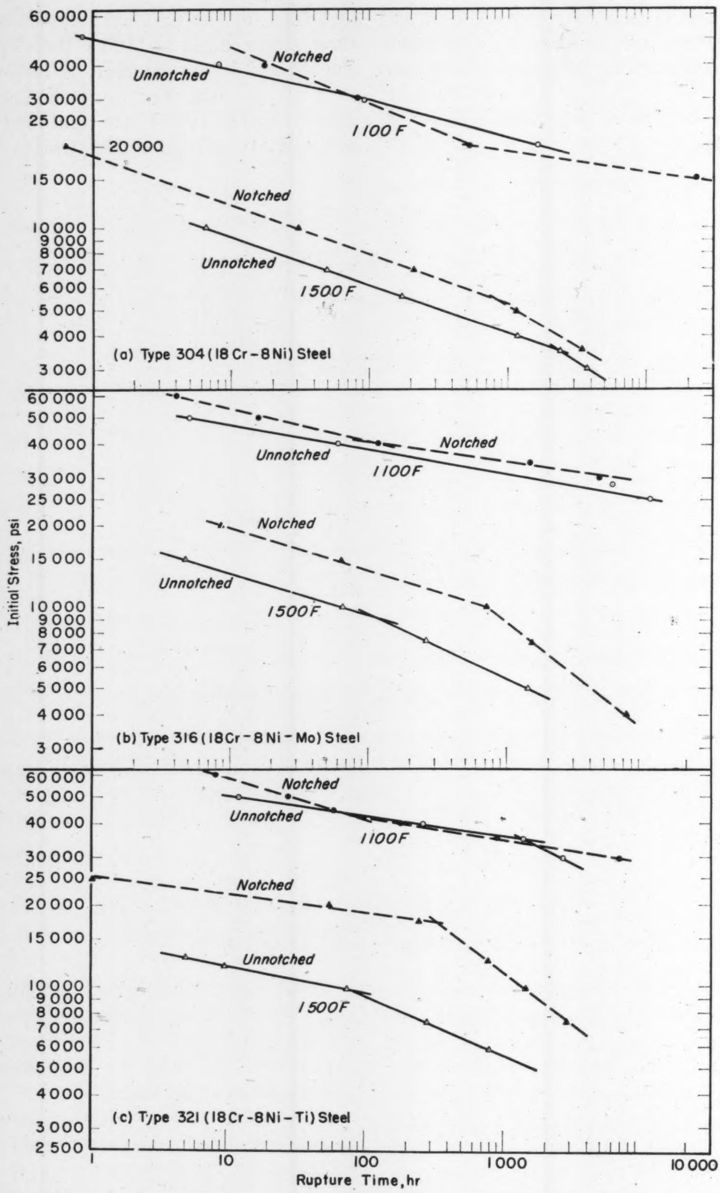


FIG. 2.—Results of Creep-Rupture Tests on Notched and Unnotched Specimens of Types 304, 316, and 321 Steel at 1100 and 1500 F.

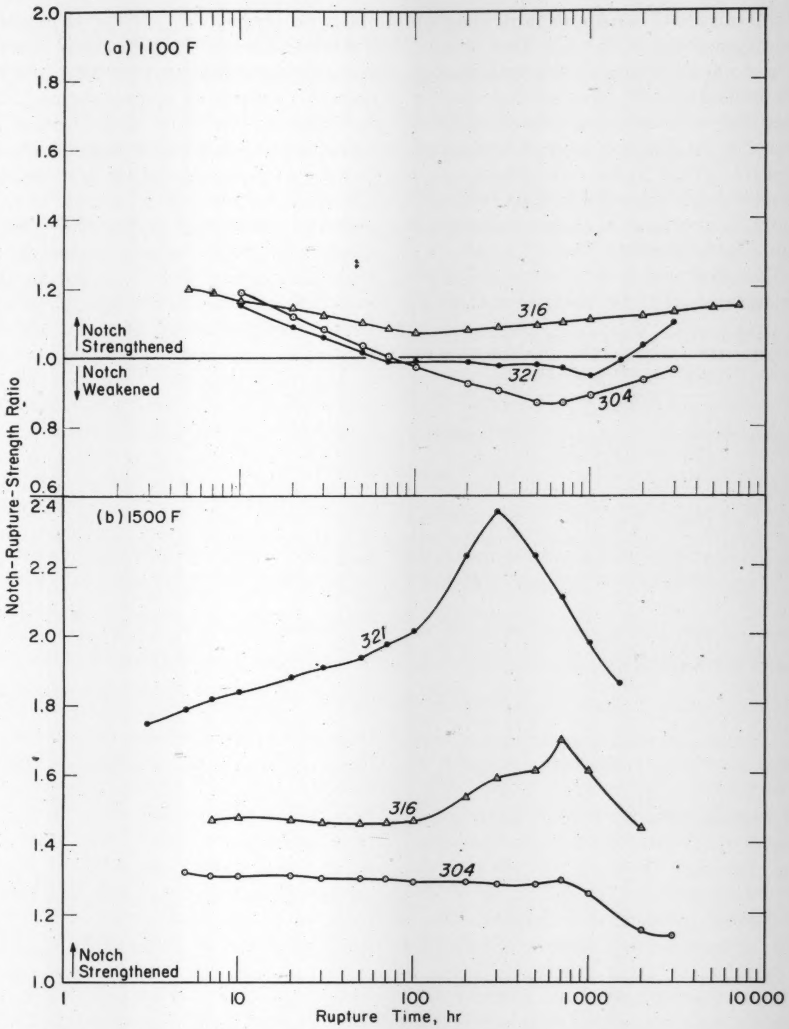


FIG. 3.—Variation of Notch Rupture-Strength Ratio with Rupture Time for Types 304, 316, and 321 Stainless Steel at 1100 and 1500 F.

The relative strengths of the three test steels at 1100 and 1500 F, and the effect of notching on each, are summarized in Table III. Types 316 and 321 have almost identical notch strength for either 100- or 1000-hr rupture life at 1100 F. As might be expected, these two steels are considerably stronger than type 304. In the unnotched condition type 321 is somewhat stronger than type 316 at

1100 F and both are appreciably stronger than type 304.

At 1500 F, the notch strength of type 321 is considerably greater than that of type 316 and more than double that of type 304. At this temperature, the creep-rupture strength for unnotched specimens is essentially identical for types 316 and 321, and both of these materials are appreciably stronger than type 304.

The effect of notching on creep-rupture strength of all three steels as a function of rupture time at 1100 F is shown in Fig. 3(*a*). To indicate the effect of notching, the ratio between rupture strength in the notched and unnotched conditions is plotted for equivalent rupture times. The curves are similar to those found for a number of other alloys (**4,7**). As is usually observed, the ratio gradually decreases with rupture time to a minimum and then increases at longer rupture times. On the basis of the present results, it seems that the minimum in the rupture-strength ratio corresponds to the point of instability in the relationship between log stress and log rupture time for either the unnotched or the notched samples. The significance of this observation is not known.

TABLE III.—COMPARISON OF CREEP-RUPTURE STRENGTH OF NOTCHED AND UNNOTCHED SAMPLES.

AISI Type	Temperature, deg Fahr	Sample Condition	Rupture Strength, psi	
			100 hr	1000 hr
304.....	1100	Notched	28 500	19 000
304.....	1100	Unnotched	29 500	21 500
304.....	1500	Notched	8 000	5 000
304.....	1500	Unnotched	6 000	4 000
316.....	1100	Notched	41 000	34 500
316.....	1100	Unnotched	38 000	31 000
316.....	1500	Notched	13 500	9 000
316.....	1500	Unnotched	9 000	5 500
321.....	1100	Notched	41 500	34 500
321.....	1100	Unnotched	42 500	36 000
321.....	1500	Notched	19 000	11 500
321.....	1500	Unnotched	9 500	5 500

Of the three steels, type 304 exhibits the lowest ratio at 1100 F, the minimum being at about 500 hr. For this material, notch strengthening is observed for rupture times up to about 70 hr. A weakening effect is found for longer rupture times; however, beyond the minimum a tendency toward strengthening is again observed. Although type 316 shows notch strengthening over the entire range of rupture times covered in this work, a minimum in the degree of strengthening is found at about 100 hr. Beyond this minimum a gradual increase in the ratio follows. Type 321 shows notch strengthening for rupture times up to about 50 hr. Beyond this point a very gradual notch-weakening effect is observed with a minimum at 1000 hr. Beyond the minimum the ratio increases and the material is notch strengthened beyond rupture times of about 1500 hr.

At 1500 F all three test steels exhibit notch strengthening over the entire range (Fig. 3(*b*)); however, the degree of strengthening does vary with rupture life and for two of the steels, types 316 and 321, goes through a maximum rather than a minimum. Type 304 shows a nearly constant notch rupture-strength ratio up to a rupture time of about 700 hr. The decrease in the ratio thereafter resulted from the inflection in the curve for notched specimens, Fig. 2. For this steel, the degree of notch strengthening may remain essentially constant for rupture times beyond about 2000 hr as the curves in Fig. 2(*a*) appear to be essentially parallel. In the type 316 material the increased ratio starting at 100 hr is due to an increase in slope of the curve for unnotched specimens. However, beyond the point of instability in the curve for notched specimens (Fig. 2(*b*)), the ratio decreases. A behavior similar to that of type 316 is also found for type 321. In general, it may be stated that beyond the point of instability in the curve for notched specimens, there is a decrease in the notch strengthening effect for types 316 and 321 steels. As may be observed from Figs. 2(*a*) and 2(*b*), this behavior is related to the fact that the slope of the second linear segment for the notched samples is greater than the slope of the second linear segment for the unnotched samples. It is not unlikely, therefore, that for very long rupture times, appreciably beyond those examined here, these materials may exhibit a notch weakening effect at 1500 F.

NATURE OF NOTCH EMBRITTLEMENT AT ELEVATED TEMPERATURES

Experimental results have been reported (**1–11**) for various commercial materials which indicate that a number of factors promote notch embrittlement at elevated temperatures. Among materials susceptible to embrittlement at elevated temperatures, in the presence of a notch, are low-alloy ferritic steels and also austenitic stainless steels. Notch embrittlement in any one material may be associated with various factors, namely, time and temperature, notch geometry (degree of stress concentration), ductility in the absence of a notch, yield strength of the material, and stress and creep-relaxation.

Effect of Time and Temperature:

The effect of time and that of temperature are interrelated and therefore it is not possible to separate the individual effect of each. It is generally found, however, that as rupture time increases, maximum notch embrittlement occurs at lower temperatures. The temperature range within which such embrittlement is observed may vary for different materials but usually extends over a span of several hundred degrees. Below and above this range, notch strengthening is usually observed. It is thus found that, for a constant rupture time, the notch-rupture-strength ratio increases with increasing temperature to a maximum, decreases sharply to a minimum, and thereafter increases again. Moreover, at any one temperature the notch-rupture-strength ratio decreases with increasing rupture time to a minimum and thereafter increases. At higher temperatures the minimum is found at shorter rupture times. The behavior found for the materials tested in the present study at 1100 F is in agreement with the behavior found by others. However, at 1500 F the variation of the notch-rupture-strength ratio differs appreciably from the general pattern.

The effect of time and temperature on the notch-rupture-strength ratio has been described (**3,4**) as a manifestation of a precipitation phenomenon. The fact that notch ductility is affected by time and temperature in a manner similar to the notch-rupture-strength ratio is offered as further substantiation of a precipitation-dependent phenomenon. In the present study, the results indicate that the behavior of the creep-rupture-strength ratio is related to some extent to the instability reflected in the relationship between the logarithm of initial stress and the logarithm of rupture time for both notched and unnotched specimens. An understanding of such instability should shed further light on the nature of notch embrittlement.

Effect of Notch Geometry:

The influence of notch geometry on notch embrittlement is usually considered in terms of the stress concentration factor, assuming elastic behavior (**12**). For relatively deep notches this factor depends on the notch depth, radius of curvature at the base of the notch, and Poisson's ratio. In general, for materials that are subject to notch embrittlement, an increase in the concentration factor causes greater embrittlement, although this is not necessarily true if the ratio of the notch radius to shank radius is changed (**8**).

In very general terms, for materials exhibiting less than 10 per cent elongation as unnotched, the presence of a mild notch usually causes notch strengthening. If a more severe notch is employed, notch embrittlement usually, but not always, results. The onset of notch embrittlement is usually defined as the point where the notch-rupture-strength ratio decreases below 1.0. As the unnotched ductility increases, however, it becomes increasingly difficult to cause notch embrittlement by increasing the severity of the notch.

As indicated in Fig. 3(*a*), types 304 and 321 exhibit notch embrittlement when exposed at 1100 F for periods between 100 hr and some much longer time. Type 321 shows less embrittlement than type 304, even though a much more severe notch was employed. It is not known whether type 316 might be notch-embrittled by employing a notch with a still higher stress concentration factor or whether the other steels might be more severely embrittled by employing a more severe notch.

Relation Between Unnotched Ductility and Notch Embrittlement:

Although materials with less than 10 per cent elongation as unnotched may tend to be embrittled in the presence of a sharp notch, the limiting ductility for the onset of notch embrittlement is not well defined. For a variety of high-temperature metals, notch embrittlement is found at relatively high unnotched-ductility levels (**3–11**). It is true, however, that the degree of notch embrittlement is usually a maximum when the unnotched ductility is at its minimum. Perhaps the presence of a notch intensifies an already existing tendency toward embrittlement. This is clearly shown by the fact that as rupture time is increased, the change in either notch-rupture-strength ratio or notch ductility parallels the change in unnotched ductility (**3,4**).

In the present study, embrittlement at 1100 F (Fig. 3(*a*)) in types 304 and 321 occurred in the rupture-time interval where the unnotched ductility is least, varying for the entire range of embrittlement between 11 and 18 per cent elongation. For type 316, notch embrittlement was not encountered even when elongations were as low as 10 per cent. However, the maximum degree of notch strengthening for this material, as for the other two at this temperature, is small. At 1500 F (Fig. 3(*b*)), the notch strengthening effect for types 316 and 321 reflects their high unnotched ductilities. For these steels, the elongation at 1500 F varied between 29 and 105 per cent. The elongation for type 304, which shows the least degree of notch strengthening at 1500 F, was appreciably less, varying between 8 and 31 per cent. It seems likely, therefore, that ductility of the unnotched specimens may influence behavior in the presence of a notch; however, insufficient data are available for a quantitative analysis.

Relationship Between Embrittlement and Yield Strength and Stress and Creep Relaxation:

A number of very interesting observations have been reported on various factors governing rupture initiation in a notched specimen (**9**). Upon loading such a specimen, high triaxial tensile stresses are developed just under the notch surface. If rupture initiation depends on the effective stress or maximum shear stress, both of which depend on notch contour, the total life of the sample may depend on the time necessary for crack initiation.

In order to delay crack initiation, it is necessary, then, to relax rapidly the triaxial stresses which are developed upon loading. Relaxation may be accomplished in two steps (**9**): first, by plastic strain just under the notch upon loading and, second, by subsequent creep relaxation. It is reported that low yield strength promotes notch strengthening because of the resulting stress relaxation which takes place by plastic deformation upon loading. Notch strengthening is also observed in materials exhibiting high rates of stress relaxation under conditions of constant strain.

It is also brought out (9) that the effective stress governs creep-rupture, therefore notch strengthening and embrittlement depend largely on the rate of relaxation of the effective stress.

MODE OF FRACTURE AND MICROSTRUCTURE

Following completion of the stress-rupture tests, several notched specimens of type 321 were prepared and stressed at 1100 F. The specimens were removed from the apparatus before rupture occurred and sectioned longitudinally for microscopic examination.

A series of photomicrographs (Figs. 4, 5, and 6) depict progress of the fracture in specimens subjected to a stress of 50,000 psi at 1100 F for 7.0, 15.1, and 22.0 hr, respectively. After 7.0 hr (Fig. 4), fracture apparently had initiated below the root of the notch and progressed intergranularly toward the notch surface and inward toward the center of the specimen. The crack is quite sharp. It is evident therefore that the mechanical notch leads to a more severe condition provided by a long sharp crack. Figures 5 and 6 show further progress

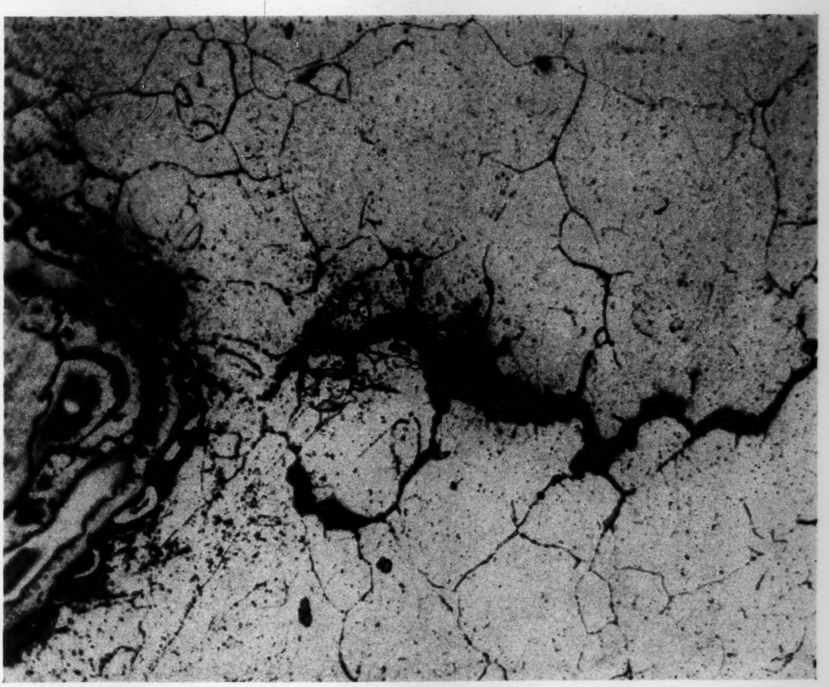


FIG. 4.—Microstructure of Notched Specimen of Type 321 Steel Subjected to a Stress of 50,000 psi for 7.0 hr at 1100 F—Vilella's Reagent (×500).

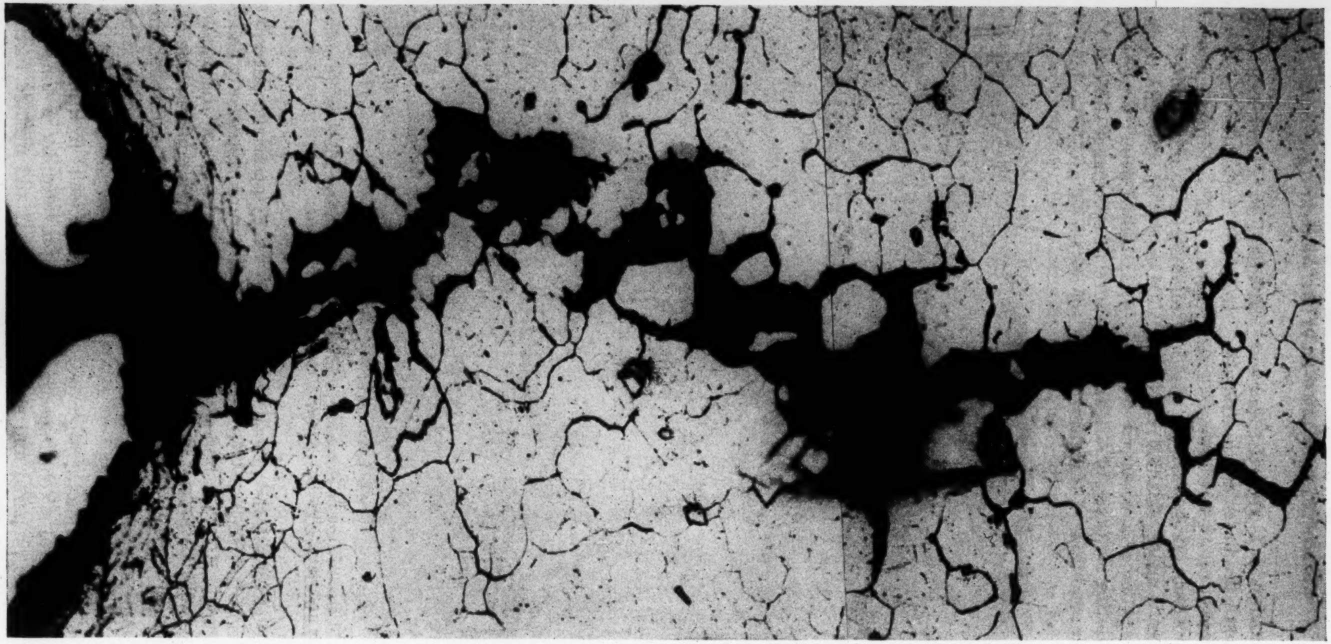


FIG. 5.—Microstructure of Notched Specimen of Type 321 Steel Subjected to a Stress of 50,000 psi for 15.1 hr at 1100 F—Vilella's Reagent (×500).

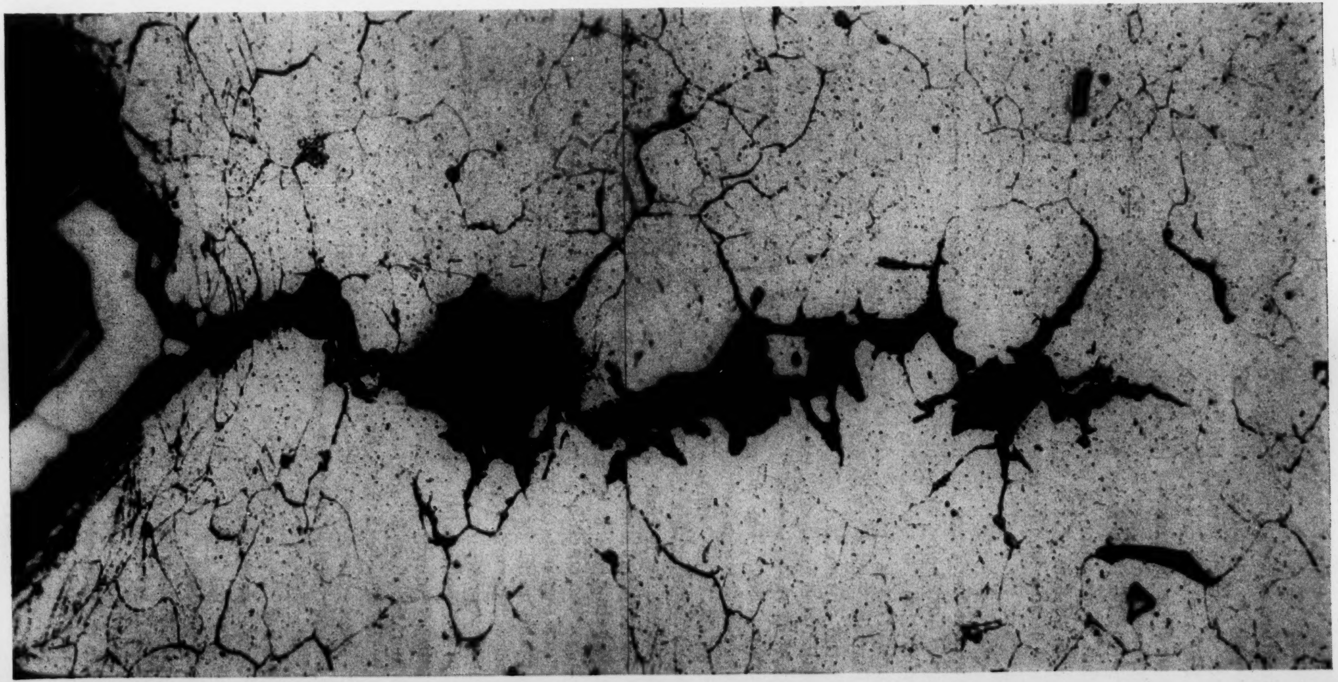


FIG. 6.—Microstructure of Notched Specimen of Type 321 Steel Subjected to a Stress of 50,000 psi for 22 hr at 1100 F—Vilella's Reagent (×500).

of the fracture which has propagated simultaneously to the notch surface and toward the specimen center. However, between 15.1 and 22 hr under load there seems to be little progress of the crack toward the specimen center. As observed by others (11) and discussed below, it appears that propagation of the crack is arrested and an appreciable amount of deformation occurs, leading to a triaxial state of stress at the center of the specimen.

The origin of the crack, as indicated in Fig. 4, lies just below the notch surface where initially the triaxiality is at a maximum. This is brought about by the fact that the radial stress decreases to zero at the surface but rises sharply inward under the notch and then decreases again as the center of the specimen is approached. The longitudinal and circumferential stresses are at a maximum at the notch surface and decrease inward, approaching the value of the radial stress. It is quite apparent from Figs. 5 and 6 that a marked opening of the propagating crack occurs; however, the tip of the crack remains sharp. Since the crack seems to be arrested it is likely that the ratio of the effective diameter below the notch to the effective radius of curvature of the notch decreases to the point where the stress concentration is much less than that existing when the test is initiated (9). After the crack has been arrested, the maximum shear stress is sufficiently high to cause appreciable creep deformation.

The initiation and propagation of rupture in specimens subjected to a stress of 40,000 psi has been found to be essentially the same as found for the specimens subjected to 50,000 psi. After 51.1 hr under a stress of 40,000 psi, a separation of the grain boundaries has been noted in the vicinity of, but slightly below, the root of the notch and again providing a very sharp crack. After 100 hr, the crack has been found to propagate outward to the notch surface and inward toward the center of the specimen. After 150 hr, the rupture had further propagated inward but it is also observed that the crack has widened and a broader external notch formed. It is again evident from these observations that the broadening of the effective notch and decrease in the effective diameter below the notch should diminish the stress concentration at the end of the crack and arrest its propagation.

After the crack is arrested following the early stages of initiation and propagation, extensive creep deformation occurs. This behavior is demonstrated clearly in Fig. 7, which shows one half of the traverse of the rupture contour of a notched specimen which failed at 1100 F in 57 hr. The one half of the specimen which is not shown is essentially a mirror image of the one half presented. This chart shows that about midway between the notch surface and specimen center, an appreciable amount of plastic deformation occurred, as evidenced by the elongated grains. In the vicinity of the center of the specimen there is again no evidence of appreciable plastic deformation. The mechanism which leads to final rupture seems to depend on a state of triaxial tensile stresses in the very center of the specimen. The triaxiality probably results from plastic constraint which dedevelops after the initial crack has been arrested. Thus, final fracture initiates at the very center of the notched specimen and propagates outward and meets the arrested crack. The behavior illustrated by Fig. 7 is typical of most notched specimens examined. Evidence of such behavior was not observed in specimens subjected to low stresses at 1100 F, which resulted in extended rupture times. In these samples, no areas of plastic

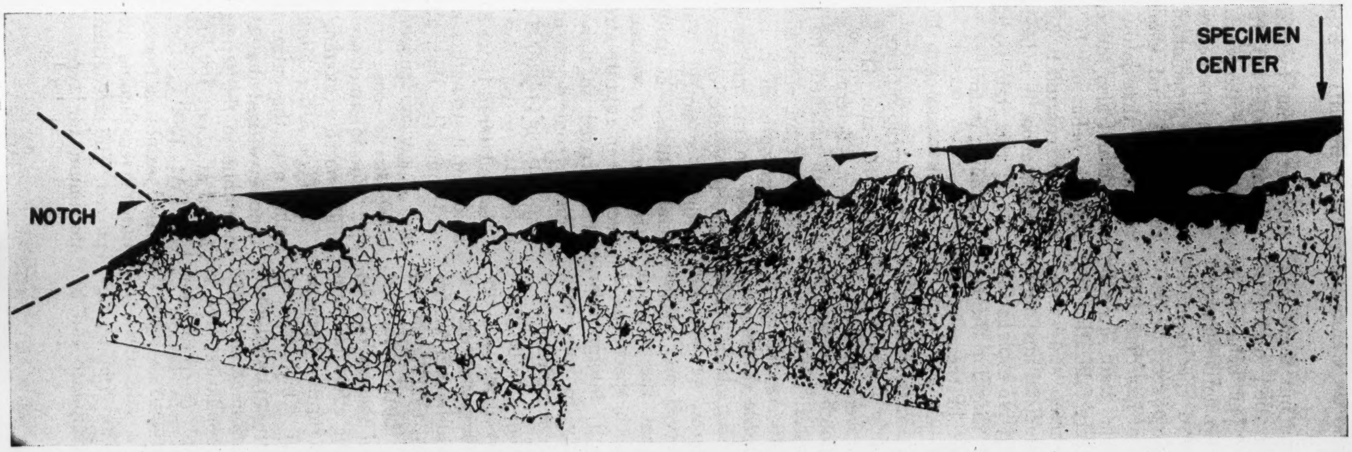

SPECIMEN CENTER
NOTCH

FIG. 7.—Traverse of Fractured Surface of Notched Specimen of Type 321 Steel Which Failed in 57.0 hr Under a Stress of 45,000 psi at 1100 F—Vilella's Reagent (×150).

deformation were found but there was evidence of recrystallization and pronounced grain growth. This behavior is not, however, limited to notched specimens. Several unnotched specimens at 1100 F showed evidence of recrystallization for rupture times of less than 1000 hr, but no appreciable grain growth seemed to occur. Recrystallization is seldom found during creep and creep-rupture testing of commercial austenitic stainless steels at 1100 F. No explanation can be offered at this time for the recrystallization observed in the present investigation.

The mode of rupture in the materials tested was predominantly intergranular whether the specimens were notched or unnotched. In the unnotched specimens tested under high stresses, the fracture seemed to initiate at the center of the specimen after appreciable deformation and pronounced necking had taken place. At lower stresses, pronounced necking was not observed and cracks were formed throughout the strained volume. Rupture of such specimens is completed when small intergranular cracks grow sufficiently to join with others causing complete separation.

Because of the predominantly intergranular-type rupture in austenitic stainless steels at elevated temperatures, it is very likely that grain-boundary behavior has an important bearing on the rupture mechanisms for both unnotched or notched specimens of these alloys. In a separate study on the effect of grain boundary precipitate on rupture-strength of a type 316 material, which will be published soon, it has been found that both size and distribution of the precipitate at the grain boundaries have a pronounced effect on creep-rupture strength at 1100 F. Normally, at 1100 F this material exhibits a thin and continuous grain-boundary precipitate. Agglomeration of this precipitate can be brought about by heating for a short time at 1300, 1400, 1500 F or to somewhat higher temperatures. A pretreatment of this type before testing at 1100 F results in a pronounced increase in both rupture life and elongation. It seems reasonable that grain boundary structure should also influence notch creep-rupture behavior. If notch embrittlement is controlled by a precipitation phenomenon, it is likely that precipitation at the grain boundaries may be a controlling factor.

SUMMARY AND CONCLUSIONS

The creep-rupture strength of types 304, 316, and 321 austenitic stainless steels was determined at 1100 and 1500 F on both notched and unnotched specimens. At an equivalent rupture time, the strength of a notched specimen, based on the net cross-sectional area, may be greater or less than that of one without a notch. Thus, at elevated temperatures, a notch may either strengthen or weaken a material. A decrease in rupture strength of notched as compared to unnotched specimens indicates some degree of notch embrittlement. In the present study the following is found:

1. At 1100 F, types 304 and 321 show mild notch-strengthening effect for rupture times less than about 100 hr and also a similar tendency beyond about 1000 hr.
2. For intermediate rupture times a slight notch-weakening effect is found.
3. Type 316 exhibits notch-strengthening over the entire range of rupture times investigated at 1100 F; however, a minimum in the strengthening effect is found at about 100 hr.
4. At 1500 F, the three materials tested exhibit a measurable degree of notch-strengthening for all stresses employed. The strengthening effect is at a maximum for types 316 and 321 at

rupture times between 300 and 700 hr; for type 304, notch strengthening was essentially constant up to 800 hr, and then decreased.

A detailed study of rupture initiation and propagation was made on type 321 material at 1100 F. Upon interrupting tests after various times under load, it was observed that rupture in notched specimens at 1100 F initiates just beneath the notch surface and then propagates to the notch surface as well as inward toward the center of the specimen. After propagating outward to the root of the notch, separation of the newly formed surfaces increases the effective radius at the root of the notch whereas the propagation of the crack inward reduces the effective specimen diameter. Thus, the stress concentration factor is decreased appreciably after initiation and propagation of the initial crack. As a result, the crack travelling inward seems to be arrested and an appreciable amount of plastic deformation follows. Such a high degree of localized plastic deformation leads to triaxial tensile stresses at the center of the specimen where rupture again initiates. Growth of the internal crack outward completes the rupture process. For the materials tested, the rupture is found to be of the intercrystalline type in all specimens examined.

In unnotched specimens at high stresses, rupture seems to begin predominantly in the center of the bar and rupture propagation occurs toward the surface. At low stresses, intergranular cracks form throughout the strained volume and total separation occurs by the propagation and joining of numerous microscopic cracks.

Acknowledgment:

The author acknowledges the participation in the study of R. R. Burt and J. H. Thomas, and of G. V. Smith who initiated the program and supervised some of the experimental work.

REFERENCES

(1) G. V. Smith, "Properties of Metals at Elevated Temperatures," McGraw-Hill Book Co., Inc., New York, N. Y. (1950).

(2) G. V. Smith and E. G. Houston, private communication.

(3) G. Sachs and W. F. Brown, Jr., "A Survey of Embrittlement and Notch Sensitivity of Heat Resisting Steels," Symposium on Effects of Notches and Metallurgical Changes on Strength and Ductility of Metals at Elevated Temperatures, Am. Soc. Testing Mats., p. 6 (1952). (Issued as separate publication *ASTM STP No. 128.*)

(4) W. F. Brown, Jr., M. H. Jones, and D. P. Newman, "Influence of Sharp Notches on the Stress-Rupture Characteristics of Several Heat-Resisting Alloys," Symposium on Effects of Notches and Metallurgical Changes on Strength and Ductility of Metals at Elevated Temperatures, Am. Soc. Testing Mats., p. 25 (1952). (Issued as separate publication *ASTM STP No. 128.*)

(5) F. C. Hull, E. K. Hann, and H. Scott, "Effect of a Notch and of Hardness on the Rupture Strength of 'Discaloy'," Symposium on Effects of Notches and Metallurgical Changes on Strength and Ductility of Metals at Elevated Temperatures, Am. Soc. Testing Mats., p. 49 (1952). (Issued as separate publication *ASTM STP No. 128.*)

(6) D. P. Newman, M. H. Jones, and W. F. Brown, Jr., "Time-Temperature Dependence of the Notch Effect and Influence of Notch Depth in Stress-Rupture Tests on a Cr-Mo-V Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 677 (1953).

(7) W. F. Brown, Jr., M. H. Jones, and D. P. Newman, "Influence of Sharp Notches on the Stress-Rupture Characteristics of Heat-Resisting Alloys: Part II," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 661 (1953).

(8) E. A. Davis and M. J. Manjoine, "Effect of Notch Geometry on Rupture Strength at Elevated Temperatures," Symposium on Effect of Notches and Metallurgical Changes on Strength and Ductility of Metals at Elevated Temperatures, Am. Soc. Testing Mats., p. 67 (1952). (Issued as separate publication *ASTM STP No. 128.*)

(9) H. R. Voorhees and J. W. Freeman, "Notch Sensitivity of Heat Resistant Alloys at Elevated Temperatures," *Technical Report 54-175*, Wright Air Development Center, Sept., 1956.

(10) J. G. Sessler and W. F. Brown, "Notch and Smooth Bar Stress-Rupture Characteristics of Several Heat-Resistant Alloys in the Temperature Range Between 600 and 1000 F," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 738 (1956).

(11) R. C. Carlson, R. J. MacDonald, and W. F. Simmons, "Factors Influencing the Notch-Rupture Strength of Heat-Resistant Alloys at Elevated Temperatures," *Transactions*, Am. Soc. Mechanical Engrs., pp. 78, 349 (1956).

(12) H. Neuber, "Kerbspannungslehre," Berlin (1937). Translation by J. Edwards, "Theory of Notch Stresses," Univ. of Michigan, Ann Arbor, Mich. (1946).

EFFECT OF ENVIRONMENT ON CREEP AND CREEP-RUPTURE BEHAVIOR OF SEVERAL STEELS AT TEMPERATURES OF 1000 TO 1200 F*

By F. Garofalo[1]

Synopsis

The creep and creep-rupture properties of several ferritic and austenitic stainless steels have been determined from tests in air, in a purified mixture of helium plus 2 per cent hydrogen and in vacuum at 1000 to 1200 F. The results indicate that the creep and creep-rupture properties are not affected by environment within the limits of the experiments performed.

The change in slope of the plot of log stress *versus* log rupture time for various steels tested in air is unchanged upon testing in vacuum or in inert atmosphere. This indicates that factors other than oxidation bring about the change in slope frequently found in such a plot.

The effect of environment on the mechanical properties of metals has received some attention. However, the experimental results are not sufficient to indicate the mechanism controlling some of the effects which have been observed.

It is somewhat surprising to find that in metals tested at room temperature the mechanical properties may depend in part on the medium surrounding the specimen. For example, in medium-carbon and low-alloy steels a slight decrease in tensile strength is found when the test specimens are surrounded by water as compared to tests in air (**1**).[2] In polycrystalline materials the effects of environment are usually small. In single crystals they seem to be appreciably more pronounced. The Rehbinder effect (**19**) in single crystals of zinc and tin is a typical example. Upon testing these crystals in contact with surface-active liquids a decrease in flow stress is found. It has been postulated (**19**) that adsorption of surface-active liquids on the glide planes results in a lowering of the resolved critical shear stress. The flow and rupture strength of single crystals of various metals are also affected by various coatings (**2,14**).

The effect of environment on elevated temperature creep and creep-rupture strength has been studied by a number of investigators (**3,15,16**). Creep-rupture tests in air, oxygen, and hydrogen indicate that for steel the mode of rupture is independent of environment (**3**). It is also reported that intergranular oxidation decreases the creep-rupture strength and therefore leads to a change in the slope of the log stress-log rupture time plot (**3**). This conclusion is based on the finding that for a low-carbon steel the change in slope is more pronounced in the presence of oxygen than in air and that for ingot iron no change in slope is found in the presence of dry hydrogen. For some materials, environment affects minimum creep rate and notch-rupture as well as creep-rupture behavior **(15,16,20,21)**.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Edgar C. Bain Laboratory for Fundamental Research, U. S. Steel Corp., Research Center, Monroeville, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Although intergranular oxidation may reduce creep-rupture strength and therefore lead to a change in slope in the plot of log stress *versus* log rupture time, other factors may be equally important in some materials. It has been found in testing aluminum that as the initial stress is lowered in successive creep tests, the ratio of grain boundary strain to over-all strain increases **(5)**. At lower stresses where a change in slope may be found, excessive grain boundary shearing may lead to lower strength and ductility. Grain boundary shearing may be related to the rate of relaxation of shearing stress at the grain boundaries. If, as is indicated by internal friction experiments **(6)**, grain boundaries behave in a viscous manner at elevated temperature, then shear-stress relaxation will lead to points of stress concentration and intergranular cracking **(6)**. This may decrease the creep strength and over-all ductility and thus lead to a change in the slope of log stress-log rupture time plot, even in the absence of an oxidation effect.

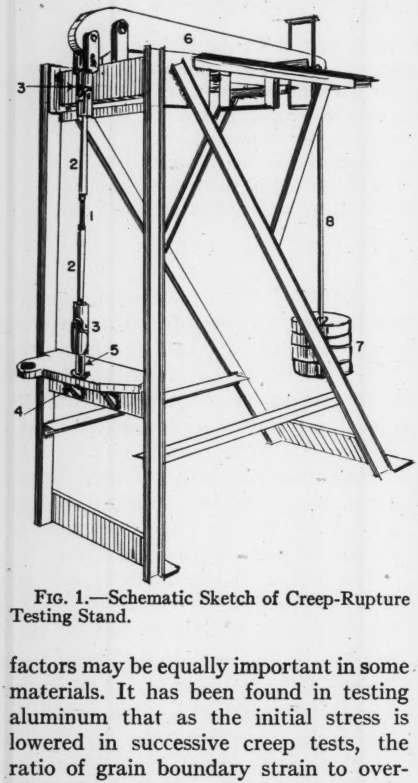


FIG. 1.—Schematic Sketch of Creep-Rupture Testing Stand.

In order to study further the effect of environment on creep and creep-rupture behavior in steels, equipment was developed for testing in purified gaseous atmospheres and in vacuum. This paper describes exploratory tests on a number of steels with this equipment.

APPARATUS

The apparatus employed in testing creep-rupture specimens in purified helium or vacuum utilizes a regular test stand and simple lever system as shown in Fig. 1. The specimen, *1*, and extension rods, *2*, are shown exposed. For testing in either helium or vacuum the extension rods form an integral part of the test chamber assembly. The universal joints, *3*, and the anchorage, a spherically seated bolt, *5*, tend to minimize nonaxial loading. Anchorage is made at *4*. The lever, *6*, gives a ratio of 9 to 1 between the tensile load on the specimen and the total weight suspended from rod *8*.

The test chamber and furnace assembly used for testing under purified helium mixture are shown in Fig. 2. The furnace utilizes a nichrome-wire heating element wound on an alundum tube. The test chamber is a 27 chromium-steel tube inserted within the alundum tube and extending beyond the top and bottom of the furnace. At the top end of the test chamber is attached a seal cup which contains a low-melting alloy.

The inverted cup attached to the upper extension rod completes the seal when the specimen assembly is inserted into the test chamber. The seal cup arrangement was designed to allow a nominal strain in the creep-rupture specimen, using a 1.0-in. gage length, of at least 100 per cent, without breaking the atmosphere. The seal at the bottom of the test chamber is formed by means of a silicone rubber gasket.

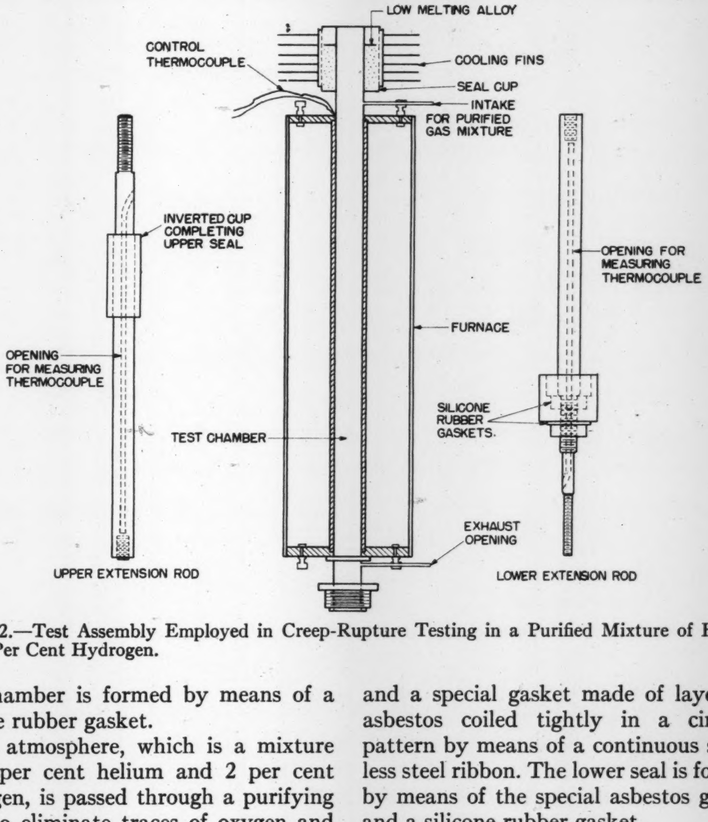

LOW MELTING ALLOY
CONTROL THERMOCOUPLE
COOLING FINS
SEAL CUP
INTAKE FOR PURIFIED GAS MIXTURE
INVERTED CUP COMPLETING UPPER SEAL
OPENING FOR MEASURING THERMOCOUPLE
FURNACE
OPENING FOR MEASURING THERMOCOUPLE
SILICONE RUBBER GASKETS.
TEST CHAMBER
EXHAUST OPENING
UPPER EXTENSION ROD
LOWER EXTENSION ROD

FIG. 2.—Test Assembly Employed in Creep-Rupture Testing in a Purified Mixture of Helium Plus 2 Per Cent Hydrogen.

The atmosphere, which is a mixture of 98 per cent helium and 2 per cent hydrogen, is passed through a purifying train to eliminate traces of oxygen and carbon dioxide. The mixture enters the test chamber at the top, is removed at the bottom and passed through a water trap. A positive flow of the gas mixture is provided throughout each experiment.

The test chamber, including the furnace assembly, employed in tests under vacuum is shown in Fig. 3. The furnace assembly is similar to the one employed in tests under helium, except that in the vacuum apparatus a window is provided for visual observation of the specimen or for extension measurements. The upper seal is formed by a flexible bellows and a special gasket made of layers of asbestos coiled tightly in a circular pattern by means of a continuous stainless steel ribbon. The lower seal is formed by means of the special asbestos gasket and a silicone rubber gasket.

The test chamber is exhausted through the opening at the bottom; the pressure within the test chamber can be measured by attaching a vacuum gage at the top opening.

A vacuum is obtained by means of a

mechanical forepump and a diffusion pump. The forepump has a capacity of 33.4 liters per min and can produce an ultimate vacuum of 5×10^{-2} μ. Forepressures of this order were not obtained with the apparatus described above. It was found that leaks at the upper and lower seals and at the observation window could not be completely eliminated and a high vacuum could not be attained within the test chamber. Forepressure could not be reduced below 20 μ and pressure within the test chamber could not be reduced below 0.2 μ.

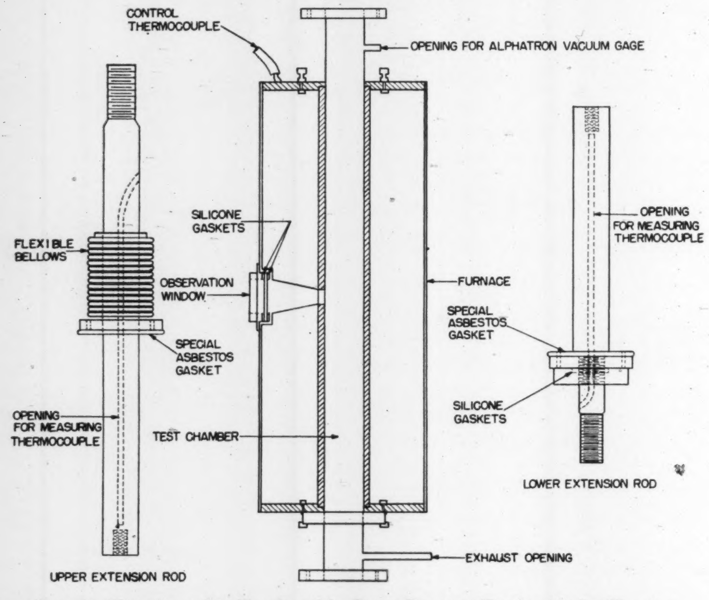

CONTROL THERMOCOUPLE
OPENING FOR ALPHATRON VACUUM GAGE
OPENING FOR MEASURING THERMOCOUPLE
SILICONE GASKETS
FLEXIBLE BELLOWS
OBSERVATION WINDOW
FURNACE
SPECIAL ASBESTOS GASKET
SPECIAL ASBESTOS GASKET
SILICONE GASKETS
OPENING FOR MEASURING THERMOCOUPLE
TEST CHAMBER
LOWER EXTENSION ROD
EXHAUST OPENING
UPPER EXTENSION ROD

FIG. 3.—Test Assembly Employed in Creep-Rupture Testing Under Vacuum.

MATERIALS

The first exploratory experiments conducted in this study were made on specimens remaining from previous test programs. In many instances only a limited number of such specimens were available for testing in purified helium-2 per cent hydrogen mixture and in vacuum for comparison with earlier results obtained by testing in air. The chemical compositions and heat treatment of the several steels studied are given in Tables I and II.

TEST RESULTS

Preliminary tests with unstressed specimens were made in the inert atmosphere and under vacuum to determine the amount of scaling to be expected in carbon, low-alloy, and stainless steels. Although carbon steels could be held at temperatures of 1000 to 1200 F in an inert atmosphere of purified helium without observable oxidation, this was not possible with stainless steels. The introduction of 2 per cent hydrogen into the helium proved effective in minimizing scaling, and produced no observable decarburization. In the vacuum apparatus it has not been possible to avoid scaling completely. The use of a titanium getter surrounding the specimen in both types of apparatus has further minimized oxidation.

Results of creep-rupture tests of a 1020 steel in air and in purified helium-2 per cent hydrogen mixture are shown in Fig. 4(*a*). These results, at 1000 and 1200 F, indicate that creep-rupture strength, at high and low stresses, is independent of environment for these tests. It is of particular importance to note that a change in slope in the log σ - log *tr* plots, where σ is the initial stress and *tr* the rupture time, is found at both temperatures. Since visible surface oxidation was prevented in all specimens tested in the purified gas mixture, whereas appreciable scaling occurred in air, it seems unlikely that for this material intergranular oxidation is responsible for the decrease in slope.

TABLE I.—CHEMICAL COMPOSITION OF TEST STEELS, PER CENT.

Steel	Type	Carbon	Manganese	Phosphorus	Sulfur	Silicone	Nickel	Chromium	Molybdenum	Nitrogen	Aluminum	Other
A......	1020	0.19	0.81	0.014	0.018	0.04	0.004	...	...	...	...	...
B....	1 chromium- ½ molybdenum	0.10	0.40	0.020	0.017	0.20	0.12	0.96	0.54	0.012	0.001	...
C......	304	0.05	0.53	0.008	0.005	0.61	10.71	18.53	...	0.028	0.005	...
D......	321	0.07	0.52	0.021	0.003	0.39	10.40	17.97	...	0.011	0.069	0.58 titanium

Test results on steel B (1 chromium-½ molybdenum) in Fig. 4(*b*) show at 1100 F a change in slope in the log σ - log *tr* beyond rupture times of 100 hr. In addition to the tests in air, tests at this temperature were made in the purified gas mixture and under vacuum. Again, creep-rupture strength seems to be independent of environment. Results at 1200 F were obtained on this steel only in the purified gas mixture. As may be observed in Fig. 4(*b*), the results fall again on two linear segments.

Creep-rupture tests of two austenitic stainless steels, types 304 and 321, were made in air and in the purified gas mixture. Results of these tests are shown in Figs. 4(*c*) and (*d*). For both of these steels, only a limited number of specimens were available for tests in the purified gas mixture. Those tests that were made in the inert gas gave creep-rupture strength values which agree well with those determined in air.

The effect of environment on minimum creep rate was determined from autographic extension-time traces (**22**). The results in Fig. 5, for all steels tested, again show that comparable results are found in air and in the purified gas mixture or under vacuum.

TABLE II.—HEAT TREATMENT OF TEST STEELS.

Steel	Type	Heat Treatment, deg Fahr
A.....	1020	1650, air cooled
B.....	1 chromium- ½ molybdenum	1700, furnace cooled
C.....	304	1900, water quenched
D....	321	1900, water quenched; 1550 for 2 hr, air cooled

It is generally assumed for steels

tested in air that the creep law describing the relationship between initial stress, σ, and minimum creep rate, *mcr*, is the so-called log-log law. This empirical relationship states that the log σ varies linearly with log *mcr*. This behavior is found in many cases for steels tested in air (**7,8**). Close examination of results in this investigation shows, however, that all steels tested exhibit two linear segments in the log σ - log *mcr* plot.

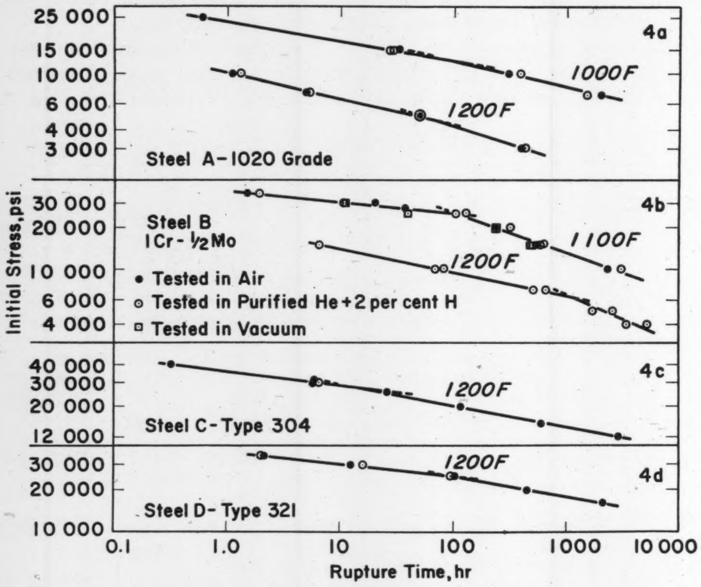


FIG. 4.—Relation Between Initial Stress and Time to Rupture for All Steels Tested.

DISCUSSION OF RESULTS

The results obtained in this study show that within the limits of materials and test conditions covered, environment has no effect on minimum creep rate nor on rupture life. As will be discussed later, it is likely that under certain conditions environment does not affect the basic modes of flow and fracture and therefore should not affect creep and creep-rupture properties.

The basic modes of flow in polycrystalline metals at elevated temperatures may differ with location within any one individual grain. In the central portion of individual grains, flow undoubtedly results from the movement of dislocations along slip planes. Because dislocation climb is more easily accomplished at elevated temperatures than at low temperatures, plastic deformation is more homogeneous, slip being distributed over a larger number of slip planes. This mechanism may lead to so-called "slipless flow" observed at stresses where slip lines are not visible. In the vicinity of grain boundaries, the mode of deformation is more inhomogeneous because of the interaction between the differently oriented grains. In these areas, folds and kink-bands are observed (**4,9,10**) as a result of each grain attempting to accommodate itself to the imposed state of stress and consequent grain boundary gliding and grain rotation.

In the over-all picture, however, the various modes of deformation must be interrelated and must depend on external stress and temperature. It is very likely that at the usual test temperatures for steels at relatively high stress, the mode of deformation is that associated with propagation of dislocations, and little grain boundary gliding (10). Deformation at high stresses in pure metals and alloys is markedly inhomogeneous and leads to observable slip lines.[3] The details of the creep mechanism at high initial stresses are not known at present (11).

At lower stresses, the creep mechanism at sufficiently high temperatures is believed to be one of dislocation climb. The relationship between minimum creep rate, *mcr*, and initial stress, σ, has been derived theoretically on this basis (11). During creep, dislocations which propagate on intersecting slip planes interact with each other and form Cottrell-Lomer dislocations. Grain boundaries and precipitated particles, as well as Cottrell-Lomer dislocations, block the propagation of free dislocations. Under these conditions, the creep rate should diminish with time throughout the creep test. However, the blocked dislocations can escape at elevated temperatures by climbing to adjacent slip planes. By estimating the rate and height of climb of dislocations between Frank-Read sources and barriers, the minimum creep rate is described by

$$mcr = C\,(\sigma^{\alpha}/RT)\exp(-Q/RT)$$

where C and α are constants, R is the

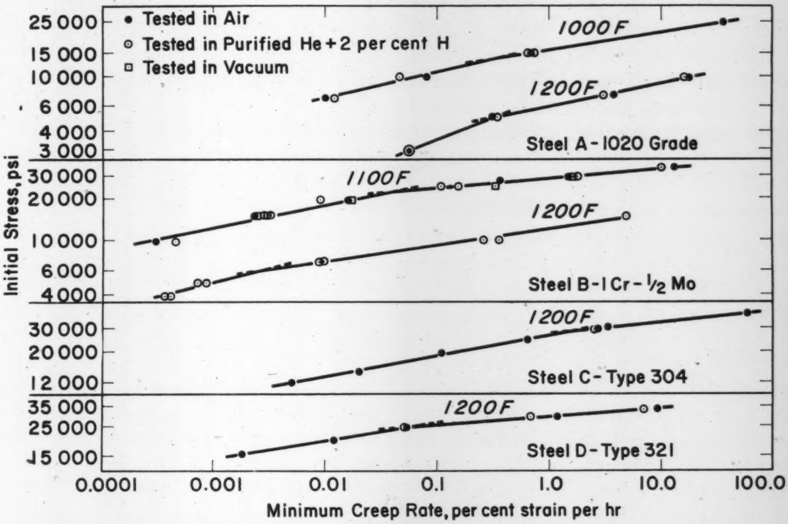


FIG. 5.—Relation Between Initial Stress and Minimum Creep Rate for All Steels Tested.

---

[3] It should be noted that a number of structural changes such as recovery, recrystallization, phase changes, and sub-boundary formation may occur at elevated temperatures and may affect the mode of deformation and the over-all creep behavior.

gas constant, T the absolute temperature, and Q is found experimentally to be equivalent to the activation energy for self-diffusion. This relation states that a linear relation exists between log *mcr* and log σ. As found in Fig. 5, the results obtained in the present study are in agreement with this relation, showing a linear relation for the log σ - log *mcr* plot at low stress levels. In many cases two linear segments are found for steels as shown in Fig. 5 and also for high-purity metals (11). As mentioned previously, the mechanism which controls the behavior at high stress levels is not understood. It is not known whether the change from the first to the second linear segment is related to a change in grain boundary behavior or the instability of Cottrell-Lomer dislocations at high stress levels. In any case, it is clear from Fig. 5 that within the limits of this study the changing of the environment from air to an inert atmosphere or vacuum has not affected creep behavior and presumably has not had any effect on the creep mechanism at low or high stresses. It would seem that at low stresses, environment should have little effect on creep behavior unless the surrounding medium readily diffuses into the test material and affects dislocation climb and propagation. Because of the effect of grain boundaries on dislocation glide, oxide films on polycrystalline specimens have little effect on creep by preventing dislocations from reaching a free surface, although such an effect may be quite pronounced in single crystals.

Because of the interrelation between minimum creep rate and rupture time (12), it is likely that if environment has no effect on creep, the rupture behavior may not be affected either. This should be true if environment does not affect tertiary creep. As shown in Fig. 4, within the limits of the present experiments, environment has not affected creep-rupture behavior. As pointed out previously, the log σ - log rupture time plots show two linear segments. The change from the first linear segment at high stress levels to the second linear segment coincides, as is usually found for steels, with the change in slope in log stress - log minimum creep rate plots. Thus the interrelation between creep and rupture is maintained (17).

Because of the interrelation between minimum creep rate and rupture life it would seem from the previous discussion that environment should have little effect on rupture behavior at high stress levels where rupture may be predominantly transgranular. The effect of environment on creep-rupture strength at low stresses, that is, along the second linear segment in Fig. 4, could be very marked. At low stresses, steels exhibit predominantly intergranular rupture, which may be related to more complete shear stress relaxation at grain boundaries and greater grain boundary flow (5,6). Thus it is possible for the surrounding medium to diffuse along the grain boundaries and have a direct effect on intergranular separation. It is noteworthy that in results of Thielemann and Parker (3) for a 1015 steel tested at 1000 F under high stresses no difference was observed between tests in air and oxygen. However, at lower stresses the more intensive intergranular oxidation in the presence of oxygen decreased the creep-rupture strength. From the results in Fig. 4 it must be concluded, however, that even when testing in air, within the limits of the present study, intergranular oxidation was not excessive and did not decrease creep-rupture strength.

SUMMARY AND CONCLUSIONS

A study was made of the creep and creep-rupture behavior of a number of ferritic and austenitic stainless steels in air, in a purified mixture of helium and 2 per cent hydrogen, and in vacuum. The experimental findings lead to the following conclusions:

1. Within the limits of the present study there is no effect of environment on either creep or creep-rupture properties.

2. At low stress levels, a linear relation is found between log stress and log minimum creep rate, which is in agreement with the dislocation climb theory. A second linear segment is found at higher stresses. If dislocation climb and propagation control the creep rate during secondary creep at low stresses, as seems likely, it follows that an effect in polycrystalline material due to environment necessitates diffusion of the surrounding medium into the material to affect dislocation climb and propagation. No such effect is indicated by the results of this study.

3. Two linear segments are found in log stress - log rupture time plots. The change from the first to the second linear segment coincides with a similar change in the log stress - log minimum creep rate plots. Thus environment has not affected the interrelation between minimum creep rate and rupture life. At high stress levels where rupture may be predominantly transgranular, no effect of environment should be expected unless the surrounding medium diffuses readily into the material to affect creep behavior and therefore rupture behavior. At low stress levels where rupture is primarily intergranular, excessive oxidation could conceivably decrease rupture life. Apparently in the present study, oxidation, when testing in air, did not decrease rupture life to any measurable degree.

Acknowledgment:

The apparatus employed in this study was built under the direction of Q. Henderson. E. G. Houston, R. M. Hood, and R. R. Keck conducted the tests. G. V. Smith supervised the initial portion of this program.

REFERENCES

(1) C. Benedicks and A. Ruben, "The Influence of Liquids Upon the Strength of Glass and Hardened Steel," *Jernkon. Ann.*, Vol. 129, p. 37 (1945). Summary in *Engineers Digest* (Am. Edition), Vol. 2, No. 10, Oct. 1945, p. 485.

(2) J. J. Gilman, "The Role of Thin Surface Films in the Deformation of Metal Monocrystals," Symposium on Basic Effects of Environment on Strength, Sealing and Embrittlement of Metals, p. 3 (1955). (Issued as separate publication *ASTM STP No. 171.*)

(3) R. H. Thielemann and E. R. Parker, "Fracture of Steels at Elevated Temperatures after Prolonged Loading," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 135, p. 559 (1939).

(4) H. C. Chang and N. J. Grant, "Inhomogeneity of Creep Deformation of Coarse-Grained High Purity Aluminum," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 197, p. 1175 (1953), and "Grain Boundary Sliding and Migration of Intercrystalline Failure Under Creep Conditions," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 197, p. 305 (1953).

(5) D. McLean, "Grain Boundary Slip During Creep of Aluminum," *Journal*, Inst. Metals, Vol. 81, p. 293 (1952–1953).

(6) C. Zener, "Elasticity and Anelasticity," Chicago University Press (1948) and "Micro-Mechanism of Fracture," *Fracturing of Metals*, Am. Soc. Metals, Cleveland, Ohio (1948).

(7) G. V. Smith and E. J. Dulis, "Creep and Rupture of Several Chromium-Nickel Austenitic Stainless Steels," *Transactions*, Am. Soc. Metals, Vol. 42, p. 935 (1950).

(8) E. J. Dulis, G. V. Smith, and E. G. Houston, "Creep and Rupture of Chromium-

Nickel Austenitic Stainless Steels," *Transactions*, Am. Soc. Metals, Vol. 45, p. 42 (1953).

(9) N. J. Grant and A. M. Gervais, "Kink Band Formation in High Purity Aluminum Under Creep Conditions," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 197, p. 1487 (1953).

(10) I. S. Servi and N. J. Grant, "Creep and Stress Rupture Behavior of Aluminum as a Function of Purity," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 191, p. 909 (1951) and "Structure Observations of Aluminum Deformed in Creep at Elevated Temperatures," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 191, p. 917 (1951).

(11) J. Weertman, "Theory of Steady State Creep Based on Dislocation Climb," *Journal of Applied Physics*, Vol. 26, No. 10, p. 1213 (1955).

(12) F. C. Monkman and N. J. Grant, "An Empirical Relationship Between Rupture Life and Minimum Creep Rate in Creep-Rupture Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 834 (1956).

(13) E. J. Dulis and G. V. Smith, "Creep and Creep-Rupture of Some Ferritic Steels Containing 5 Per Cent to 17 Per Cent Chromium," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 627 (1953).

(14) L. C. Weiner and M. Gensamer, "Effects of Solid Environments on the Brittle Fracture of Zinc Single Crystals," *Journal*, Inst. Metals, Vol. 85, p. 441 (1956–1957).

(15) Paul Shahinian, "Effect of Environment on Creep-Rupture Properties of Some Commercial Alloys," *Transactions*, Am. Soc. Metals, Vol. 49, p. 862 (1957).

(16) E. D. Sweetland and E. R. Parker, "Effect of Surface Condition on Creep of Some Commercial Metals," *Journal of Applied Mechanics*, Vol. 20, No. 30, March 1953.

(17) F. Garofalo, Discussion of paper by F. C. Monkman and N. J. Grant, "An Empirical Relationship Between Rupture Life and Minimum Creep Rate in Creep-Rupture Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 593 (1956).

(18) L. A. Shepard and W. T. Giedt, "Transgranular and Intergranular Fracture of Ingot Iron during Creep," *Technical Notes 4285*, Nat. Advisory Committee for Aeronautics, Aug., 1958.

(19) P. A. Rehbinder, V. P. Lichman, and V. M. Maslennikos, "Deformation of Single Crystals of Metals as Facilitated by Adsorption of Surface Active Substances," *Comptes Rendus*, Académie des Sciences, N. R., Section Sciences, Vol. 32, p. 125 (1941).

(20) O. C. Shepard and W. Schalliol, "The Effect of Environment on the Stress-Rupture Properties of Metals at Elevated Temperatures," Symposium on Corrosion of Materials at Elevated Temperatures, p. 34 (1950). (Issued as separate publication *ASTM STP No. 108*.)

(21) P. Shahinian and M. R. Achter, "The Effect of Atmosphere on Creep-Rupture Properties of A Nickel-Chromium-Aluminum Alloy," *NRL Report 5133*, Naval Research Laboratory, May 6, 1958.

(22) G. V. Smith, W. G. Benz, and R. F. Miller, "Creep and Creep-Rupture Testing," *Proceedings*, Am. Soc. Testing Mats, Vol. 47, p. 615 (1947).

DISCUSSION

Mr. John Rittenhouse.[1]—Would the author care to comment on the degree of vacuum and also what the leak rate was in this vacuum test?

Mr. F. Garofalo (*author*).—As explained in the paper the vacuum was only of the order of 0.2 μ. As far as the leak rate is concerned I can only give you a rough estimate. We certainly did not have a static vacuum. It is estimated it would take of the order of about 10 hr for the pressure to climb back to atmospheric pressure.

Mr. Herbert T. Corten.[2]—The author has considered the question of mechanism: Has he determined the activation energies associated with these processes as an aid in identifying the mechanism?

Mr. Garofalo.—There are two reasons why activation energies were not calculated from the data obtained. The first is that the tests were made under conditions of constant load, and since the actual stress on the specimen is a variable such calculations could not be made. Secondly, the steels tested are complex alloys and the meaning of an activation energy could not be clearly defined.

Mr. T. E. Tietz.[3]—One simple approach for determining the activation energy for creep in the case of a constant load test is to change the temperature rapidly during test and determine the creep rate just prior to the temperature change and immediately after the temperature change. Using this technique, no assumption is necessary regarding the existence of identical work-hardened states of the material, and the activation energy may be calculated directly from the two strain rates corresponding to the two test temperatures.

Mr. Garofalo.—It is true that this could be done, assuming a very small change in stress occurred during creep at the two temperatures. But again it should be pointed out that since the steels tested are very complex materials it would be extremely difficult to define the meaning of such an activation energy.

Chairman H. C. Cross.[4]—I wonder if you would refresh my memory. Is your work in agreement, or does it show some different behavior from what I recall possibly Sheppard out at Stanford showed in a paper some eight years ago? Did he not show an effect from oxygen as compared with vacuum, or am I mistaken?

Mr. Garofalo.—As indicated in the printed paper there has been some evidence showing that such effects are found. However, in our case similar effects were not found. One of the reasons our results do not indicate an effect of environment within the limits of our tests is perhaps that the temperatures employed were fairly low. Thus the effects of oxidation would be less in our case than in the case of others who investigated somewhat higher temperatures.

---

[1] Research Specialist, Materials Section, California Institute of Technology, Jet Propulsion Laboratory, Pasadena, Calif.

[2] Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[3] Metallurgy and Ceramics, Lockheed Missile and Space Division, Palo Alto, Calif.

[4] Metallurgist and Assistant Coordination Director, Battelle Memorial Institute, Columbus, Ohio.

But in any event this would mean that if an effect is found, particularly on minimum creep rate, one must visualize some mechanism by which the dislocation behavior is affected.

MR. MARION SEMCHYSHEN.[5]—In a paper by P. Shahinian and M. R. Achter,[6] of the U. S. Naval Research Laboratory, presented at the Sixty-first Annual Meeting of ASTM, it was concluded that the test atmosphere did affect the creep-rupture properties of a nickel-chromium-aluminum allow. In a subsequent paper which appears in the *Transactions* of the American Society for Metals,[7] these same authors again found that creep and rupture properties of a nickel-chromium alloy were affected by the ambient atmosphere. Other workers have concluded that the test atmosphere affects creep and rupture properties of other metallic materials.

Would the author of the paper please comment on why the results reported here appear to contradict those of other workers?

MR. GAROFALO.—It is not possible to give a satisfactory answer to this question at this time. It should be mentioned, however, that it is likely that at temperatures higher than those investigated in the present study an effect of environment might be found. Perhaps Mr. Shahinian, who is present at this meeting, might have something to add to this.

MR. PAUL SHAHINIAN.[8]—Most of the materials that we studied were nickel-base alloys, and I believe this is the work just referred to. We did find large differences between air and vacuum tests, especially at long times. We also did a small amount of work on steels—I think tests were run at only one or two stress levels at a single temperature—and we found small differences between air and vacuum test specimens.

---

[5] Supervisor, Climax Molybdenum Company of Michigan, Detroit, Mich.

[6] The Effect of Atmosphere on Creep-Rupture Properties of a Nickel-Chromium-aluminum Alloy, *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 761 (1958).

[7] Vol. 51, p. 244.

[8] Metallurgist, U. S. Naval Research Laboratory, Washington, D. C.

EFFECT OF NOTCH GEOMETRY ON TENSILE PROPERTIES OF ANNEALED TITANIUM AT 100, 25, −78, AND −196 C*

By G. W. Geil[1] and N. L. Carwile[1]

Synopsis

The combined effects of low temperatures and geometry of a circumferential notch on the tensile behavior of initially annealed commercially pure titanium were investigated. The notch geometry was varied by changes in its depth and root radius; the notch depth, in terms of the cross-sectional area removed in machining the notch, ranged from 5 to 87 per cent; the root radius ranged from 0.005 to 2.0 in. True stress - true strain data were obtained to the initiation of the fracture of specimens at 100, 25, −78, and −196 C, and representative true stress - true strain curves are presented. The relationships of selected strength and ductility indices to the notch geometry and to the accompanying triaxialities and stress concentration factors are presented graphically and discussed in some detail.

The brittle failures at low temperatures of metals that ordinarily show good ductility in tension at room temperature often cause considerable concern regarding the significance of the tension tests. It is now generally recognized that the ductility of a metal as measured by a tension test is significant only for conditions equivalent to those under which the test is made. Brittle failures in service are generally associated with notches that are ordinarily present in the structure either as design features (threads, keyways, and so on) or defects (deep scratches, weld flaws, and grinding marks). Consequently, it is necessary to evaluate the tensile behavior of metals not only by ordinary tension tests but also by tests that take into account the presence of notches, their shape and size.

Many investigations have been made to determine the tensile properties of unnotched specimens of titanium at room and subzero temperatures. However, only limited data are available on the combined effects of notches and low temperatures on the tensile behavior of this metal. Most of the published data on notched specimens of titanium were obtained either in tests at different temperatures on specimens with a constant notch geometry **(1–5)**[2] or in tests at room temperature on specimens of selected notch depths **(6,7)**. Results obtained at the National Bureau of Standards in tension tests at a series of low temperatures on unnotched and notched specimens (circumferential V-notches of 60-

---

*** Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.**

[1] National Bureau of Standards, Washington, D. C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

deg angle, selected notch depths, and a constant root radius of 0.05 in.) were summarized in a previous paper (8). The experimental investigation was expanded to determine the combined effects of low temperature, notch depth, root radius, and the accompanying stress concentrations and induced triaxial stresses on the tensile behavior of specimens of the same lot of titanium. The results obtained in the latter phase of the study are summarized in the present paper.

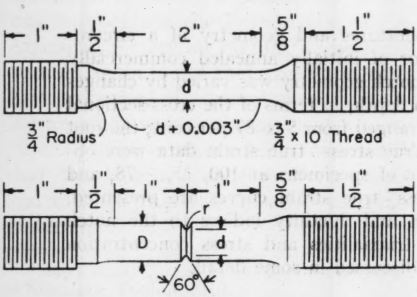


FIG. 1.—Unnotched and Notched Specimens Used in the Tension Tests.

MATERIAL, SPECIMENS, AND TEST PROCEDURES

Material:

Test specimens were prepared from bars processed from a single heat of commercially pure titanium (ASTM Specification B 265, grade 4). The 1-in. round bars were supplied as hot rolled and annealed for a fine grain size (ASTM grain size No. 9). The impurities determined in the titanium were as follows:[3] oxygen, 0.21; nitrogen, 0.04; hydrogen, 0.012; carbon, 0.04; silicon, 0.02; iron, W; chromium, W; tin, W; aluminum, VW; magnesium, VW; manganese, VW; nickel, VW; and copper, T.

Specimens:

The specimens were of circular cross-section (Fig. 1). The unnotched specimens had a reduced section that was gradually tapered; the diameter at the midsection (0.400 in.) was approximately 0.003 in. less than that at the ends of the reduced section. The notched specimens were machined with a circumferential V-notch of 60-deg angle and a constant minimum diameter, *d*, of 0.350 in. at the root of the notch. The notch depth[4] ranged from 5 to 87 per cent and was obtained by proper selection of the diameter, *D*, of the cylindrical portion of the specimen adjacent to the notch. The radius at the root of the notch was machined to selected values ranging from 0.005 to 2.0 or 10 in. The shape of the circumferential V-notch degenerated into a simple groove whose profile was an arc of a circle as the root radius was increased to relatively large values or as the notch depth was decreased to relatively small values. For the notches included in this investigation, the groove profiles were arcs of a circle as follows:

Notch depth of 87 per cent, root radius of 2.0 in.,
Notch depth of 70 per cent, root radius of 0.5 in. or greater,
Notch depth of 50 per cent, root radius of 0.2 in. or greater,
Notch depth of 30 per cent, root radius of 0.1 in. or greater,
Notch depth of 10 per cent, root radius of 0.025 in. or greater, and
Notch depth of 5 per cent, root radius of 0.01 in. or greater.

As the unnotched specimens were machined with a slight taper over the reduced gage length, these specimens may be considered as notched specimens in which the notches are approximately arcs of about 300 in. radius.

---

[3] Numerical values are in per cent by weight. Letters represent relative ranges of weight as determined by spectrographic analysis: W, weak (0.01 to 0.1 per cent); VW, very weak (0.001 to 0.01 per cent); and T, trace (0.0001 to 0.001 per cent). The spectrographic method used was not sensitive for determination of tungsten.

[4] Notch depth is expressed as the per cent of the cross-sectional area removed in machining the notch in the specimen; it is equal to 100 $(D^2 - d^2)/D^2$.

Test Procedures:

A detailed description of the tension test equipment and the method of maintaining the selected temperature is given in previous publications (9, 10, 11). The tension tests were made at temperatures of 100, 25, −78, and −196 C with the unnotched and notched specimens in a pendulum hydraulic testing machine. The specimens were strained slowly to fracture at a deformation rate of about 0.5 to 1.0 per cent contraction in area per min during the deformation beyond initial yielding. The tests above and below room temperature were made employing a constant temperature bath in which the specimens were immersed 30 min prior to and during the application of the load. Simultaneous load and diameter (minimum diameter of unnotched specimens, diameter at base of the notch of the notched specimens) measurements were made throughout the entire test. The diameter measurements were made with a special reduction of area gage (9) designed for use at low temperatures.

RESULTS AND DISCUSSION

Tensile Data:

The data obtained in tension tests at 100, 25, −78, and −196 C on both the unnotched and notched specimens are summarized in Table I. The headings of some of the columns will be defined later in the text in the discussions of these properties.

With the equipment used, it was not feasible to obtain autographic load-strain curves. Thus, no yield strength values are reported in the table. However, the values reported for the true stress[5] at a true strain[6] (elastic plus plastic) of 0.025 give an indication of the stresses at which appreciable yielding has occurred. The amount of plastic strain included in the 0.025 true strain was not constant as the elastic strain, in general, tended to increase with (1) increase in the notch depth, (2) decrease in the root radius, and (3) decrease in the temperature. Some of the deep-notched specimens with small root radii retained very little ductility at −78 and −196 C and fractured at true strains less than 0.025.

True Stress - True Strain Relations:

Representative true stress - true strain curves for specimens tested in tension at 100, 25, −78, or −196 C are presented in Figs. 2 to 8. Logarithmic graphs (not shown) of the true stress - true strain data for uniaxial tension (extension of unnotched specimens to the maximum load) were significantly sigmoidal. Thus, these data do not conform to any simple relationship, such as the frequently employed relation $\sigma = K\delta^n$, where σ and δ are the true stress and true strain, respectively, and K and n are constants.

The notch geometry, with its accompanying stress concentrations and induced triaxiality, had a very pronounced effect on the resistance to flow and ductility of the specimens. The resistance to flow, in general, increased with an increase in notch depth (Figs. 2 to 5) or true strain and decreased with an increase in root radius (Figs. 2 to 8) or temperature (Figs. 3, 6, 7, and 8).

At each temperature, the increase in resistance to flow with increase in notch depth was relatively small as the depth

---

[5] True stress was determined by dividing the current load by the current minimum cross-sectional area of the specimen.

[6] True strain was determined as the natural logarithm of the ratio of the initial minimum cross-sectional area of the specimen, A_0, to the current minimum cross-sectional area, A.

TABLE 1.—TENSILE PROPERTIES OF UNNOTCHED AND NOTCHED (60-DEG ANGLE) SPECIMENS OF INITIALLY ANNEALED COMMERCIALLY PURE TITANIUM.

Notch Depth, per cent	D,[a] in.	d,[b] in.	Root Radius, r, in.	Stress Concentration Factor, K_t	Triaxiality,[c] S_t/S_l	Tensile Strength or Notch[d] Strength, psi	Ratio of Notch Strength to Tensile Strength	True Stress in psi at			True Strain[e] at		Reduction of Area, per cent	Relative[f] Energy of Deformation at Fracture	Location[g] of Fracture Initiation
								True[e] Strain of 0.025	Maximum Load	Initial Fracture	Maximum Load	Initial Fracture			
							TENSION TESTS AT +100 C								
0[h]	...	0.400	...	1.0	0.00	75 000	...	66 000	85 000	116 000	0.13	0.77	57	1.00	A
5	0.360	0.350	0.01	2.3	0.03	76 000	1.01	64 000	87 000	110 000	0.14	0.55	38	0.65	R
5	0.360	0.350	0.05	1.6	0.03	77 000	1.03	68 000	87 000	119 000	0.12	0.63	49	0.83	R
10	0.370	0.350	0.01	2.7	0.08	80 000	1.07	69 000	92 000	108 000	0.15	0.38	30	0.45	R
10[h]	0.370	0.350	0.05	1.7	0.08	82 000	1.09	73 000	94 000	122 000	0.13	0.53	40	0.66	R
10	0.370	0.350	0.1	1.4	0.08	83 000	1.11	73 000	95 000	134 000	0.13	0.69	50	0.92	R
10	0.370	0.350	0.2	1.3	0.08	83 000	1.11	72 000	96 000	134 000	0.14	0.74	53	0.92	A
10	0.370	0.350	0.5	1.1	0.08	84 000	1.12	73 000	99 000	131 000	0.16	0.69	50	0.94	A
10	0.370	0.350	2.0	1.03	0.06	81 000	1.08	68 000	93 000	127 000	0.14	0.70	52	0.94	A
30	0.418	0.350	0.01	3.5	0.22	97 000	1.29	81 000	113 000	115 000	0.15	0.18	15	0.22	R
30	0.418	0.350	0.05	1.9	0.22	98 000	1.30	84 000	116 000	130 000	0.15	0.33	30	0.52	R
30	0.418	0.350	0.1	1.6	0.20	97 000	1.29	87 000	113 000	144 000	0.16	0.59	45	0.92	R
30	0.418	0.350	0.2	1.3	0.18	92 000	1.23	79 000	108 000	138 000	0.16	0.61	48	0.92	A
50	0.495	0.350	0.005	5.3	0.35	110 000	1.47	100 000	122 000	123 000	0.10	0.12	10	0.16	R
50	0.495	0.350	0.01	3.9	0.35	114 000	1.52	101 000	128 000	129 000	0.12	0.13	12	0.17	R
50	0.495	0.350	0.025	2.7	0.34	111 000	1.48	100 000	127 000	134 000	0.14	0.21	19	0.32	R
50[h]	0.495	0.350	0.05	2.0	0.32	108 000	1.44	96 000	128 000	142 000	0.16	0.34	29	0.61	R
50	0.495	0.350	0.1	1.6	0.28	103 000	1.37	92 000	120 000	144 000	0.15	0.50	41	0.82	A
50	0.495	0.350	0.2	1.3	0.23	96 000	1.28	86 000	110 000	137 000	0.14	0.53	43	0.83	A
50	0.495	0.350	0.5	1.1	0.15	90 000	1.20	81 000	90 000	131 000	0.15	0.56	45	0.84	A
50	0.495	0.350	2.0	1.03	0.07	79 000	1.05	67 000	93 000	125 000	0.15	0.69	56	0.96	A
70	0.639	0.350	0.01	4.1	0.44	130 000	1.73	113 000	146 000	147 000	0.11	0.13	12	0.22	R
70[h]	0.639	0.350	0.05	2.1	0.35	119 000	1.59	104 000	137 000	154 000	0.14	0.37	31	0.68	A
87	0.990	0.350	0.005	5.8	0.48	139 000	1.85	123 000	150 000	151 000	0.08	0.09	8	0.14	R
87	0.990	0.350	0.01	4.2	0.47	140 000	1.87	120 000	156 000	160 000	0.11	0.14	13	0.25	R
87	0.990	0.350	0.025	2.8	0.44	134 000	1.79	111 000	156 000	166 000	0.14	0.25	23	0.46	R
87	0.990	0.350	0.05	2.1	0.40	125 000	1.67	111 000	143 000	157 000	0.14	0.32	29	0.58	A
87	0.990	0.350	0.1	1.6	0.33	111 000	1.48	96 000	128 000	143 000	0.14	0.37	33	0.61	A
87	0.990	0.350	0.2	1.3	0.24	98 000	1.30	88 000	115 000	134 000	0.16	0.46	40	0.70	A
87	0.990	0.350	0.5	1.2	0.15	86 000	1.15	73 000	101 000	131 000	0.16	0.62	48	0.88	A
87	0.990	0.350	2.0	1.03	0.07	83 000	1.11	75 000	95 000	127 000	0.14	0.67	50	0.91	A

TENSION TESTS AT +25 C

TENSION TESTS AT +25 C

0[h]	...	0.400	...	1.00	0.00	94 000	...	86 000	107 000	145 000	0.13	0.71	52	1.00	A
5[h]	0.360	0.350	0.01	2.3	0.03	96 000	1.02	88 000	110 000	121 000	0.13	0.28	25	0.35	R
5	0.360	0.350	0.05	1.6	0.03	99 000	1.05	94 000	112 000	140 000	0.12	0.49	41	0.63	R
5	0.360	0.350	0.2	1.2	0.03	98 000	1.04	87 000	113 000	152 000	0.14	0.65	48	1.02	A
5	0.360	0.350	2.0	1.03	0.03	97 000	1.03	83 000	117 000	152 000	0.18	0.64	46	1.00	A
10	0.370	0.350	0.005	3.5	0.08	103 000	1.10	83 000	123 000	127 000	0.17	0.21	16	0.26	R
10	0.370	0.350	0.01	2.7	0.08	102 000	1.09	90 000	121 000	126 000	0.17	0.22	17	0.26	R
10	0.370	0.350	0.025	2.1	0.08	103 000	1.10	88 000	122 000	136 000	0.17	0.30	24	0.40	R
10[h]	0.370	0.350	0.05	1.7	0.08	104 000	1.11	94 000	122 000	141 000	0.16	0.36	32	0.50	R
10	0.370	0.350	0.1	1.4	0.08	104 000	1.11	90 000	122 000	153 000	0.16	0.53	42	0.79	R
10	0.370	0.350	0.2	1.3	0.08	103 000	1.10	93 000	121 000	154 000	0.16	0.54	47	0.86	A
10	0.370	0.350	0.5	1.1	0.08	103 000	1.10	91 000	121 000	152 000	0.16	0.57	45	0.85	A
10	0.370	0.350	2.0	1.03	0.06	103 000	1.10	90 000	118 000	148 000	0.16	0.60	46	0.89	A
30	0.418	0.350	0.005	4.7	0.22	120 000	1.28	105 000	...	131 000	...	0.09	7	0.11	R
30	0.418	0.350	0.01	3.5	0.22	118 000	1.26	104 000	...	136 000	...	0.14	10	0.18	R
30	0.418	0.350	0.025	2.5	0.22	123 000	1.31	102 000	147 000	147 000	0.17	0.17	14	0.24	R
30[h]	0.418	0.350	0.05	1.9	0.22	123 000	1.31	110 000	146 000	155 000	0.17	0.25	23	0.38	R
30	0.418	0.350	0.1	1.6	0.20	120 000	1.28	108 000	142 000	165 000	0.17	0.45	38	0.75	R
30	0.418	0.350	0.2	1.3	0.18	117 000	1.24	104 000	138 000	161 000	0.17	0.47	39	0.75	A
30	0.418	0.350	0.5	1.1	0.13	110 000	1.17	100 000	129 000	152 000	0.16	0.47	40	0.72	A
30	0.418	0.350	2.0	1.03	0.07	102 000	1.09	93 000	118 000	149 000	0.14	0.56	45	0.81	A
50	0.495	0.350	0.005	5.3	0.35	144 000	1.53	133 000	...	150 000	...	0.06	4	0.08	R
50[h]	0.495	0.350	0.01	3.9	0.35	144 000	1.53	124 000	...	156 000	...	0.08	8	0.13	R
50	0.495	0.350	0.025	2.7	0.34	146 000	1.55	136 000	166 000	167 000	0.13	0.14	12	0.24	R
50[h]	0.495	0.350	0.05	2.0	0.32	141 000	1.50	130 000	164 000	174 000	0.15	0.22	22	0.46	R
50	0.495	0.350	0.1	1.6	0.28	134 000	1.43	124 000	156 000	176 000	0.15	0.36	31	0.59	A
50	0.495	0.350	0.2	1.3	0.23	123 000	1.31	115 000	140 000	161 000	0.14	0.36	32	0.59	A
50	0.495	0.350	0.5	1.1	0.15	111 000	1.18	102 000	127 000	150 000	0.14	0.42	37	0.64	A
50	0.495	0.350	2.0	1.03	0.07	102 000	1.09	94 000	118 000	148 000	0.14	0.55	45	0.81	A
50	0.495	0.350	10.0	1.0	0.03	97 000	1.03	85 000	114 000	146 000	0.16	0.61	47	0.88	A
70	0.639	0.350	0.01	4.1	0.44	167 000	1.78	151 000	...	180 000	...	0.08	7	0.14	R
70	0.639	0.350	0.025	2.7	0.39	166 000	1.77	122 000	188 000	195 000	0.14	0.17	16	0.32	R
70[h]	0.639	0.350	0.05	2.1	0.35	159 000	1.69	138 000	180 000	193 000	0.13	0.23	23	0.44	A
70	0.639	0.350	0.1	1.6	0.29	138 000	1.47	122 000	158 000	171 000	0.14	0.25	23	0.42	A
70	0.639	0.350	0.2	1.3	0.24	125 000	1.33	110 000	147 000	163 000	0.16	0.34	31	0.56	A
70	0.639	0.350	0.5	1.2	0.15	108 000	1.15	97 000	127 000	155 000	0.16	0.50	40	0.76	A
70	0.639	0.350	2.0	1.03	0.07	100 000	1.06	89 000	119 000	150 000	0.17	0.57	44	0.81	A
87	0.990	0.350	0.005	5.8	0.48	166 000	1.77	149 000	...	174 000	...	0.05	4	0.08	R
87	0.990	0.350	0.01	4.2	0.47	174 000	1.85	151 000	...	189 000	...	0.08	7	0.15	R
87	0.990	0.350	0.025	2.8	0.44	166 000	1.77	143 000	196 000	201 000	0.16	0.19	16	0.38	R
87[h]	0.990	0.350	0.05	2.1	0.40	161 000	1.71	140 000	183 000	194 000	0.13	0.21	20	0.41	A
87	0.990	0.350	0.1	1.6	0.33	143 000	1.52	129 000	166 000	174 000	0.15	0.22	21	0.42	A
87	0.990	0.350	0.2	1.3	0.24	125 000	1.33	115 000	147 000	162 000	0.16	0.32	29	0.56	A
87	0.990	0.350	0.5	1.2	0.15	110 000	1.17	100 000	129 000	151 000	0.16	0.44	38	0.66	A
87	0.990	0.350	2.0	1.03	0.07	96 000	1.02	91 000	117 000	147 000	0.15	0.57	46	0.81	A

---

[a] Diameter of cylindrical portion of the specimen adjacent to the notch.

[b] Minimum diameter of unnotched specimen or diameter of notched specimen at the base of the notch.

[c] Triaxiality is determined as the ratio of transverse stress to longitudinal stress and is based on the method of Sachs and Lubahn (13).

[d] Notch strength determined by dividing the highest load attained by the original minimum cross-sectional area.

[e] Includes elastic and plastic deformation.

[f] Determined as the ratio of the areas under the true stress - true strain curves of the notched and unnotched specimens, respectively.

[g] R, fracture initiated at or near the root of the notch; A, fracture initiated at or near the axis of the specimen.

[h] Tensile values are the average of two or more specimens.

[Continued on pages 990 and 991.]

TABLE I.—*Concluded.*

Notch Depth, per cent	D,[a] in.	d,[b] in.	Root Radius, r, in.	Stress Concentration Factor, K_t	Triaxiality,[c] S_t/S_l	Tensile Strength or Notch[d] Strength, psi	Ratio of Notch Strength to Tensile Strength	True Stress in psi at			True Strain[e] at		Reduction of Area, per cent	Relative[f] Energy of Deformation at Fracture	Location[g] of Fracture Initiation
								True[e] Strain of 0.025	Maximum Load	Initial Fracture	Maximum Load	Initial Fracture			
							TENSION TESTS AT −78 C								
0	...	0.400	...	1.0	0.00	124 000	...	115 000	146 000	179 000	0.16	0.52	42	1.00	A
5	0.360	0.350	0.01	2.3	0.03	128 000	1.03	119 000	...	145 000	...	0.12	11	0.18	R
5	0.360	0.350	0.05	1.6	0.03	128 000	1.03	119 000	154 000	162 000	0.18	0.25	24	0.43	R
10	0.370	0.350	0.01	2.7	0.08	128 000	1.03	121 000	...	141 000	...	0.10	9	0.15	R
10	0.370	0.350	0.05	1.7	0.08	134 000	1.08	122 000	164 000	164 000	0.20	0.20	19	0.34	R
10	0.370	0.350	0.2	1.3	0.08	132 000	1.06	119 000	160 000	187 000	0.19	0.42	36	0.81	A
10	0.370	0.350	2.0	1.03	0.06	128 000	1.03	116 000	158 000	182 000	0.21	0.43	34	0.81	A
30	0.418	0.350	0.01	3.5	0.22	146 000	1.18	148 000	...	151 000	...	0.03	4	0.04	R
30	0.418	0.350	0.05	1.9	0.22	156 000	1.26	144 000	...	177 000	...	0.13	12	0.25	R
30	0.418	0.350	0.1	1.6	0.20	153 000	1.23	139 000	186 000	196 000	0.20	0.26	22	0.53	R
30	0.418	0.350	0.2	1.3	0.18	147 000	1.19	135 000	180 000	198 000	0.20	0.34	29	0.71	A
50	0.495	0.350	0.005	5.3	0.35	159 000	1.28	...	...	162 000	...	0.02	2	0.02	R
50	0.495	0.350	0.01	3.9	0.35	170 000	1.37	168 000	...	175 000	...	0.03	3	0.05	R
50	0.495	0.350	0.025	2.7	0.34	181 000	1.46	159 000	...	193 000	...	0.07	7	0.13	R
50	0.495	0.350	0.05	2.0	0.32	177 000	1.43	155 000	...	200 000	...	0.12	10	0.25	R
50	0.495	0.350	0.1	1.6	0.28	168 000	1.35	151 000	208 000	208 000	0.21	0.21	19	0.48	A
50	0.495	0.350	0.2	1.3	0.23	158 000	1.27	144 000	184 000	188 000	0.15	0.18	18	0.36	A
50	0.495	0.350	0.5	1.1	0.15	139 000	1.12	126 000	170 000	182 000	0.20	0.28	25	0.46	A
50	0.495	0.350	2.0	1.03	0.07	128 000	1.03	118 000	159 000	179 000	0.21	0.39	34	0.75	A
70	0.639	0.350	0.01	4.1	0.44	185 000	1.49	179 000	...	191 000	...	0.03	4	0.06	R
70	0.639	0.350	0.05	2.1	0.35	199 000	1.61	170 000	...	223 000	...	0.12	12	0.26	A
87	0.990	0.350	0.005	5.8	0.48	161 000	1.30	...	...	163 000	...	0.01	1	0.01	R
87	0.990	0.350	0.01	4.2	0.47	193 000	1.56	...	...	198 000	...	0.02	3	0.04	R
87	0.990	0.350	0.025	2.8	0.44	215 000	1.73	190 000	...	232 000	...	0.07	7	0.18	R
87	0.990	0.350	0.05	2.1	0.40	200 000	1.61	180 000	...	217 000	...	0.09	10	0.19	A
87	0.990	0.350	0.1	1.6	0.33	180 000	1.45	172 000	...	194 000	...	0.07	9	0.15	A
87	0.990	0.350	0.2	1.3	0.24	160 000	1.21	149 000	...	184 000	...	0.14	15	0.28	A
87	0.990	0.350	0.5	1.2	0.15	140 000	1.13	127 000	169 000	184 000	0.19	0.30	26	0.58	A
87	0.990	0.350	2.0	1.03	0.07	128 000	1.03	118 000	156 000	180 000	0.20	0.42	35	0.81	A

TENSION TESTS AT −196 C

0	...	0.400	...	1.00	0.00	169 000	...	164 000	200 000	228 000	0.17	0.33	30	1.00	A
5	0.360	0.350	0.01	2.3	0.03	169 000	1.00	170 000	...	177 000	...	0.05	5	0.10	R
5	0.360	0.350	0.05	1.6	0.03	177 000	1.05	173 000	...	199 000	...	0.12	11	0.33	R
5	0.360	0.350	0.5	1.2	0.03	175 000	1.04	172 000	208 000	217 000	0.18	0.22	21	0.64	R
5	0.360	0.350	2.0	1.03	0.03	174 000	1.03	172 000	205 000	214 000	0.17	0.22	20	0.63	A
10	0.370	0.350	0.005	3.5	0.08	177 000	1.05	173 000	...	183 000	...	0.03	3	0.04	R
10	0.370	0.350	0.01	2.7	0.08	175 000	1.04	177 000	...	180 000	...	0.03	3	0.04	R
10	0.370	0.350	0.025	2.1	0.08	179 000	1.06	178 000	...	189 000	...	0.05	5	0.10	R
10	0.370	0.350	0.05	1.7	0.08	183 000	1.08	182 000	...	199 000	...	0.08	8	0.18	R
10	0.370	0.350	0.1	1.4	0.08	182 000	1.08	175 000	...	203 000	...	0.11	10	0.29	R
10	0.370	0.350	0.2	1.3	0.08	183 000	1.08	175 000	216 000	216 000	0.16	0.16	15	0.45	R
10	0.370	0.350	0.5	1.1	0.08	182 000	1.08	175 000	214 000	221 000	0.17	0.20	19	0.62	A
10	0.370	0.350	2.0	1.03	0.06	177 000	1.05	173 000	206 000	213 000	0.15	0.19	18	0.57	A
30	0.418	0.350	0.005	4.7	0.22	169 000	1.00	...	...	172 000	...	0.02	1	0.02	R
30	0.418	0.350	0.01	3.5	0.22	189 000	1.12	...	...	194 000	...	0.02	1	0.03	R
30	0.418	0.350	0.025	2.5	0.22	204 000	1.21	194 000	...	211 000	...	0.03	2	0.07	R
30	0.418	0.350	0.05	1.9	0.22	213 000	1.26	215 000	...	221 000	...	0.04	5	0.11	R
30	0.418	0.350	0.1	1.6	0.20	209 000	1.24	198 000	...	226 000	...	0.08	7	0.23	R
30	0.418	0.350	0.2	1.3	0.18	202 000	1.20	196 000	...	228 000	...	0.13	13	0.38	A
30	0.418	0.350	0.5	1.1	0.13	190 000	1.12	187 000	...	208 000	...	0.09	10	0.25	A
30	0.418	0.350	2.0	1.03	0.07	179 000	1.06	173 000	212 000	218 000	0.17	0.20	19	0.42	A
50	0.495	0.350	0.005	5.3	0.35	150 000	0.89	...	...	151 000	...	0.01	1	0.02	R
50	0.495	0.350	0.01	3.9	0.35	197 000	1.17	...	...	200 000	...	0.02	1	0.03	R
50	0.495	0.350	0.025	2.7	0.34	232 000	1.37	235 000	...	239 000	...	0.03	3	0.08	R
50	0.495	0.350	0.05	2.0	0.32	246 000	1.46	233 000	...	255 000	...	0.05	5	0.15	R
50	0.495	0.350	0.1	1.6	0.28	234 000	1.38	226 000	...	250 000	...	0.06	8	0.20	A
50	0.495	0.350	0.2	1.3	0.23	214 000	1.27	207 000	...	226 000	...	0.06	7	0.15	A
50	0.495	0.350	0.5	1.1	0.15	195 000	1.15	194 000	...	207 000	...	0.06	8	0.17	A
50	0.495	0.350	2.0	1.03	0.07	181 000	1.07	174 000	210 000	212 000	0.15	0.16	14	0.45	A
70	0.639	0.350	0.01	4.1	0.44	205 000	1.21	...	...	209 000	...	0.02	2	0.02	R
70	0.639	0.350	0.025	2.7	0.39	236 000	1.40	236 000	...	242 000	...	0.03	3	0.05	R
70	0.639	0.350	0.05	2.1	0.35	258 000	1.53	243 000	...	270 000	...	0.05	5	0.13	A
70	0.639	0.350	0.1	1.6	0.29	236 000	1.40	224 000	...	248 000	...	0.05	6	0.14	A
70	0.639	0.350	0.2	1.3	0.24	216 000	1.28	216 000	...	226 000	...	0.04	6	0.11	A
70	0.639	0.350	0.5	1.2	0.15	192 000	1.14	191 000	...	202 000	...	0.06	8	0.14	A
70	0.639	0.350	2.0	1.03	0.07	178 000	1.05	176 000	208 000	208 000	0.16	0.16	16	0.45	A
87	0.990	0.350	0.005	5.8	0.48	124 000	0.73	...	...	125 000	...	0.01	1	0.01	R
87	0.990	0.350	0.01	4.2	0.47	208 000	1.23	...	...	211 000	...	0.01	2	0.02	R
87	0.990	0.350	0.025	2.8	0.44	234 000	1.38	...	...	238 000	...	0.02	2	0.03	A
87	0.990	0.350	0.05	2.1	0.40	257 000	1.52	246 000	...	265 000	...	0.03	4	0.12	A
87	0.990	0.350	0.1	1.6	0.33	240 000	1.42	232 000	...	250 000	...	0.04	5	0.11	A
87	0.990	0.350	0.2	1.3	0.24	214 000	1.27	214 000	...	222 000	...	0.04	5	0.09	A
87	0.990	0.350	0.5	1.2	0.15	192 000	1.14	189 000	...	204 000	...	0.06	8	0.16	A
87	0.990	0.350	2.0	1.03	0.07	176 000	1.04	172 000	208 000	218 000	0.17	0.22	21	0.66	A

was increased from 70 to 87 per cent, as illustrated in Figs. 4 and 5 for specimens tested at 25 C; this small change was even less prominent in the tests made at −78 or −196 C. Similarly, the increase in resistance to flow with decrease in root radius was relatively small as the radius was decreased below 0.05 in. As is illustrated for the 50 per cent notch depth specimens tested at 25 C (Fig. 3), the change in the resistance to flow was considerably greater with change in root radius from 0.5 to 0.05 in. than from 0.05 to 0.005 in. The relatively small change in resistance to flow with increase in notch depth from 70 to 87 per cent or decrease in radius below 0.05 in. is associated with the relatively small

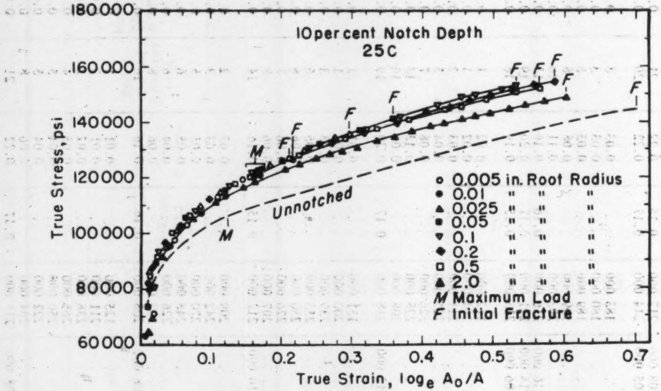


FIG. 2.—True Stress - True Strain Relationships Obtained in Tests at 25 C on Specimens of 10 per cent Notch Depth.

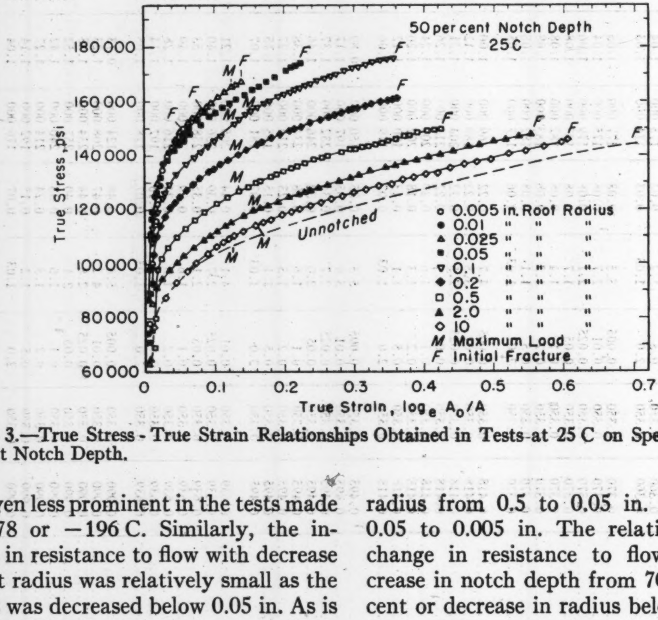


FIG. 3.—True Stress - True Strain Relationships Obtained in Tests at 25 C on Specimens of 50 per cent Notch Depth.

increase in triaxiality with the above changes in notch geometry; this feature is discussed below in relation to Fig. 9.

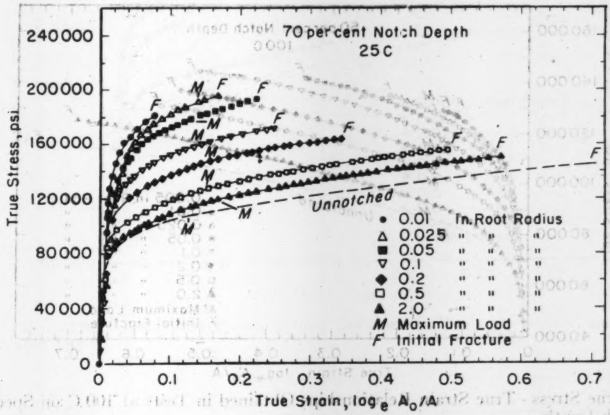


FIG. 4.—True Stress - True Strain Relationships Obtained in Tests at 25 C on Specimens of 70 per cent Notch Depth.

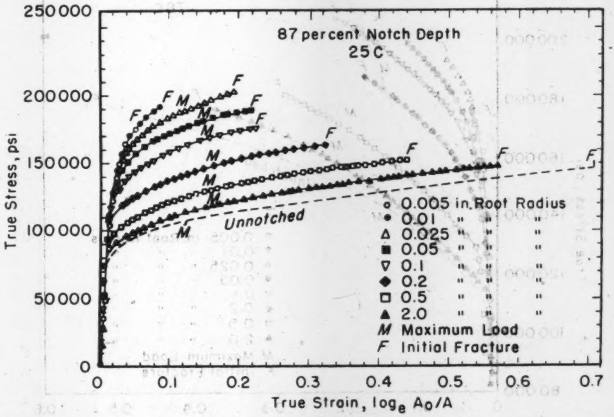


FIG. 5.—True Stress - True Strain Relationships Obtained in Tests at 25 C on Specimens of 87 per cent Notch Depth.

Triaxiality and Stress Concentration Factor:

Triaxiality.—The triaxiality, S_t/S_l, is based on the method proposed by Sachs and Lubahn (13); that is, $S_t/S_l = 1 - \frac{1}{R}$, where S_t is the transverse stress, S_l is the longitudinal stress and R is the ratio of notch strength to the tensile strength. Thus, the triaxiality is an average value of this ratio over the minimum cross-section of the specimen. Notch strength was determined by dividing the highest load attained by the initial minimum cross-sectional area at the notch. In determining the values of R, the notch strength values are restricted to those in which the specimen had sufficient ductility to attain a normal maximum load condition; that is, the slope of the load-extension curves became zero. The triaxiality depends primarily upon the notch geometry, and the values reported are averages for each specific notch geometry. For the notched specimens that did not attain a normal maximum load condition at low tem-

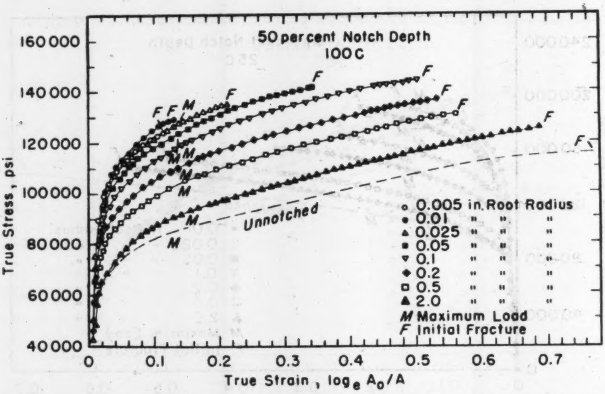


FIG. 6.—True Stress - True Strain Relationships Obtained in Tests at 100 C on Specimens of 50 per cent Notch Depth.

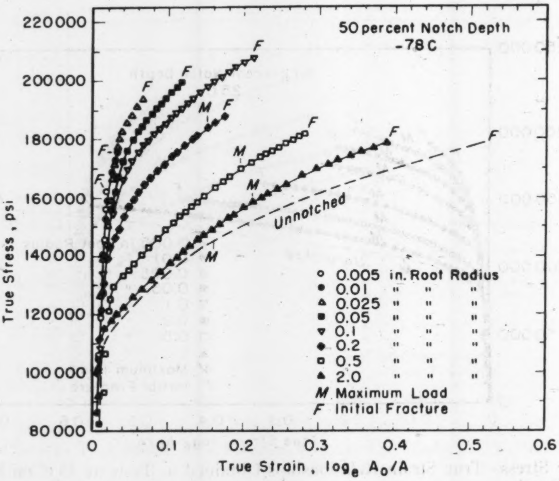


FIG. 7.—True Stress - True Strain Relationships Obtained in Tests at −78 C on Specimens of 50 per cent Notch Depth.

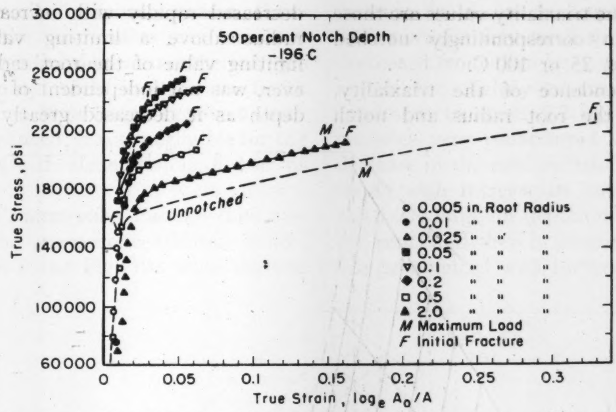


FIG. 8.—True Stress - True Strain Relationships Obtained in Tests at −196 C on Specimens of 50 per cent Notch Depth.

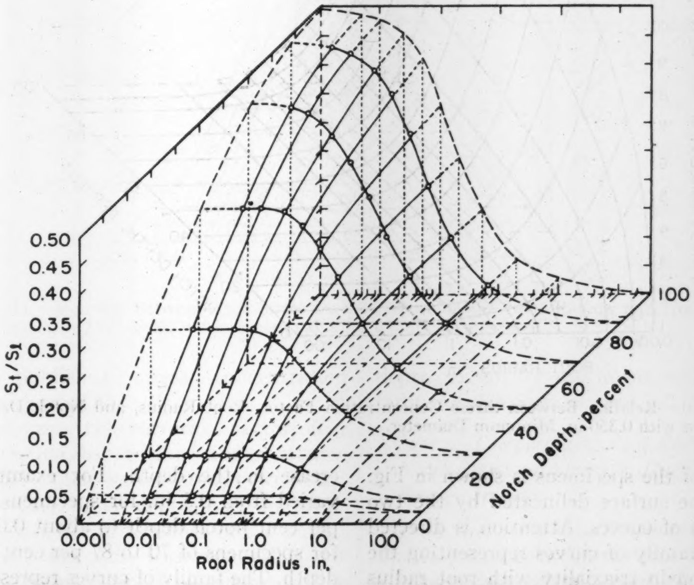


FIG. 9.—Relation Between Triaxiality, Root Radius, and Notch Depth for Specimens with 0.350 in. Minimum Diameter.

peratures, the triaxiality values are those obtained on correspondingly notched specimens at 25 or 100 C.

The dependence of the triaxiality, S_t/S_l, on the root radius and notch depth of the specimens is shown in Fig. 9 by the surface delineated by the two families of curves. Attention is directed to the family of curves representing the variation in triaxiality with root radius for constant notch depths. (Note that the root radius is plotted on a logarithmic scale in this and following figures.) The triaxiality remained nearly constant for small values of the root radius and then decreased rapidly with increase in the radius above a limiting value. This limiting value of the root radius, however, was not independent of the notch depth as it decreased greatly with increase in the depth. For example, it varied from 0.5 in. for specimens of 10 per cent notch depth to about 0.005 in. for specimens of 70 to 87 per cent notch depth. The family of curves representing variation in triaxiality with notch depth for a constant root radius show a nearly uniform increase in triaxiality with increase in notch depth for specimens with a root radius equal to or less than the

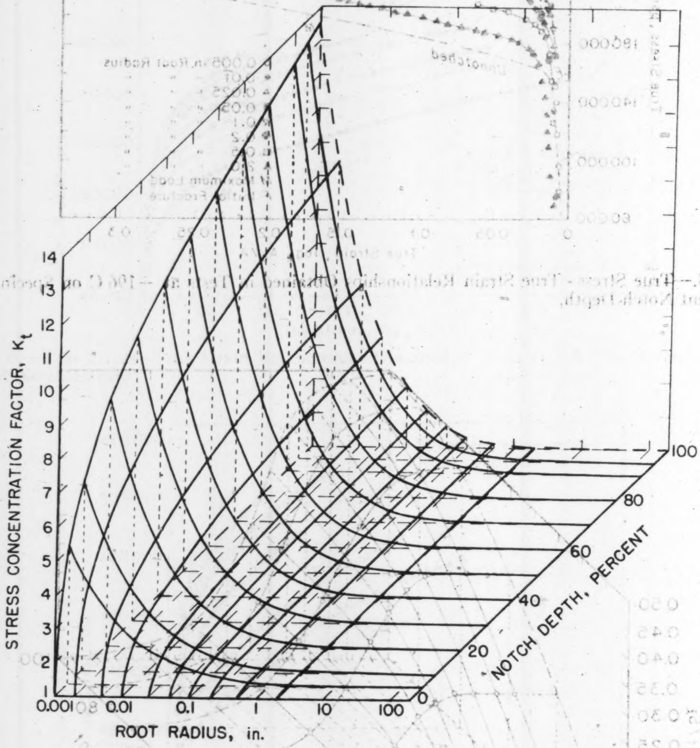


FIG. 10.—Relation Between Stress Concentration Factor, Root Radius, and Notch Depth for Specimens with 0.350 in. Minimum Diameter.

above described limiting values. However, as the root radius was increased above these limiting values the increase in triaxiality with increase in notch depth above 20 or 30 per cent was much less pronounced; it was negligible for the specimens with a root radius of 2 in. or greater.

Stress Concentration Factor.—The values for the stress concentration factor, K_t (Table I and Fig. 10), were derived from published stress concentration design charts (12).

The stress concentration factor, K_t, for a round bar with a single circumferential notch varies greatly with the root radius, notch depth, and the minimum diameter at the base of the notch. The variation of K_t with root radius and notch depth for tensile specimens with a circumferential notch of 60-deg angle and a constant minimum diameter of 0.350 in. is reported in Table I and shown in Fig. 10 by the surface delineated by the two families of curves. The increase in K_t with decrease in root radius is relatively small as the radius is decreased from that (approximately 300 in.) of the slightly tapered unnotched specimens to about 0.2 in. However, it becomes very pronounced with further decrease in the root radius. The increase in K_t with increase in notch depth is relatively large for depths up to 20 or 30 per cent, and then it generally becomes less pronounced with further increase in depth, except for specimens with a root radius of 0.005 in. or less.

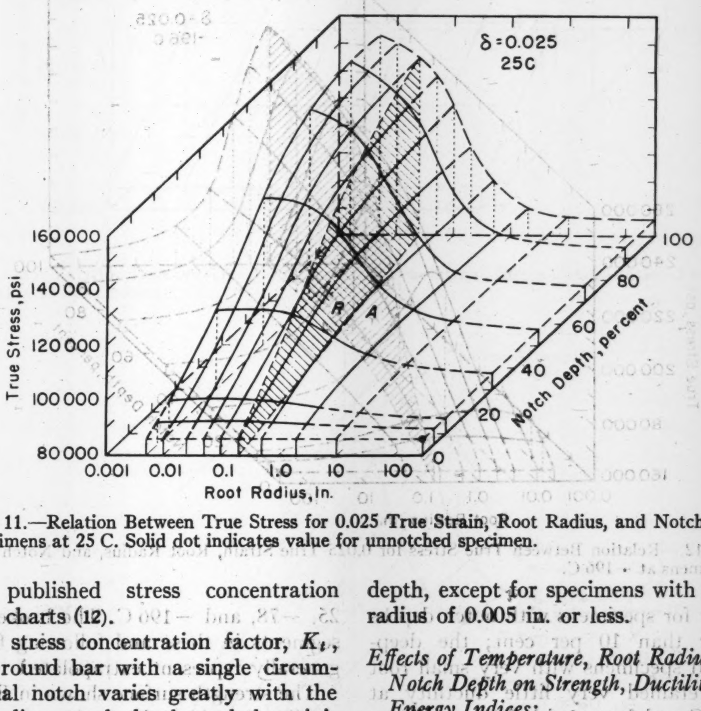


FIG. 11.—Relation Between True Stress for 0.025 True Strain, Root Radius, and Notch Depth of Specimens at 25 C. Solid dot indicates value for unnotched specimen.

Effects of Temperature, Root Radius, and Notch Depth on Strength, Ductility, and Energy Indices:

True Stress at a True Strain of 0.025.—The relations between the true stress at a true strain (elastic plus plastic) of 0.025 and the root radius and notch depth are illustrated by the surfaces delineated in Figs. 11 and 12 for the specimens tested at 25 and −196 C, respectively. The corresponding strength surfaces (not shown) obtained for the specimens at 100 and −78 C were very similar to the one at 25 C except that the corresponding stress values at 100 C were approximately 20 per cent lower, and those at −78 C were approximately 35 per cent higher than those at 25 C. The strength surface representing the test data obtained at −196 C (Fig. 12) is limited to root radii of 0.025 in. or greater for specimens with notch depths greater than 10 per cent; the deep-notched specimens with very small root radii retained very little ductility at −196 C and fractured before reaching a true strain of 0.025.

The hatched vertical sections shown in Figs. 11 and 12 and in many of the following figures represent the boundary between the specimens, *A*, in which the fracture was initiated at or near the axis, and those, *R*, in which the fracture was initiated at or near the root of the notch. The position of the initiation of the fracture of the titanium specimens was readily determined from visual observation of the fracture surfaces or photographs of these surfaces taken at 7× magnification using slightly oblique illumination. The chevron patterns pointing back to the origin of the fracture were generally very distinct, especially for the specimens fractured at 25, −78, and −196 C. The broken-line segments in these and following figures generally represent extrapolated values.

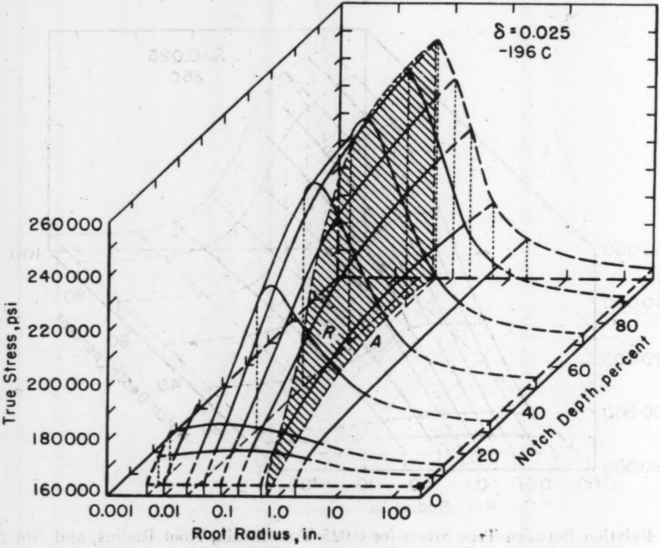


FIG. 12.—Relation Between True Stress for 0.025 True Strain, Root Radius, and Notch Depth of Specimens at −196 C.

The strength surface shown in Fig. 11 closely resembles the surface shown in Fig. 9 for the variation of the triaxiality with the notch geometry; this indicates a direct correlation of this strength index with the induced triaxiality. This relationship is clearly illustrated in Fig. 13 which shows the variation in the true stress at a true strain of 0.025 with the triaxiality and stress concentration fac-

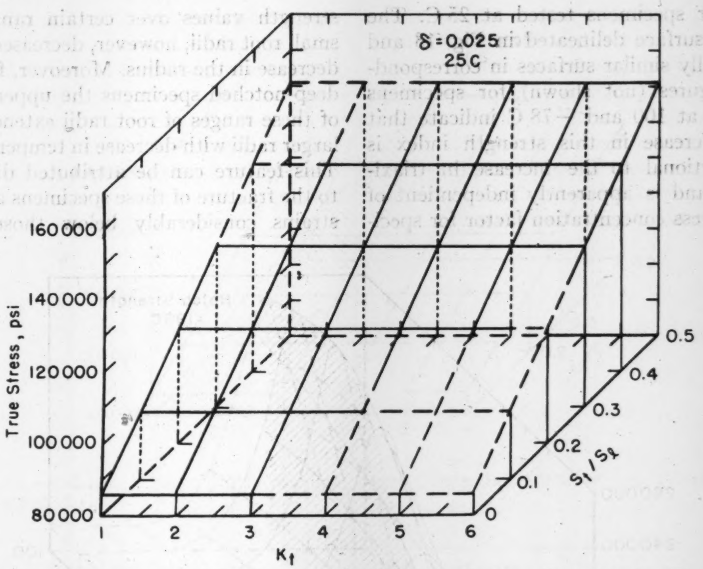


FIG. 13.—Relation Between True Stress for 0.025 True Strain, Stress Concentration Factor, and Triaxiality of Specimens at 25 C.

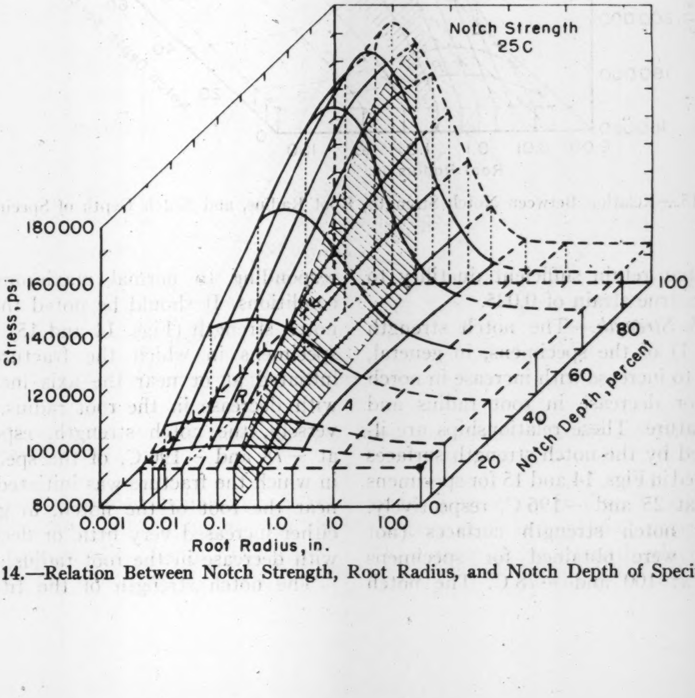


FIG. 14.—Relation Between Notch Strength, Root Radius, and Notch Depth of Specimens at 25 C.

tor for specimens tested at 25 C. The plane surface delineated in Fig. 13 and generally similar surfaces in corresponding figures (not shown) for specimens tested at 100 and −78 C indicate that the increase in this strength index is proportional to the increase in triaxiality and is apparently independent of the stress concentration factor for specimens that retain sufficient ductility to attain a true strain of 0.025.

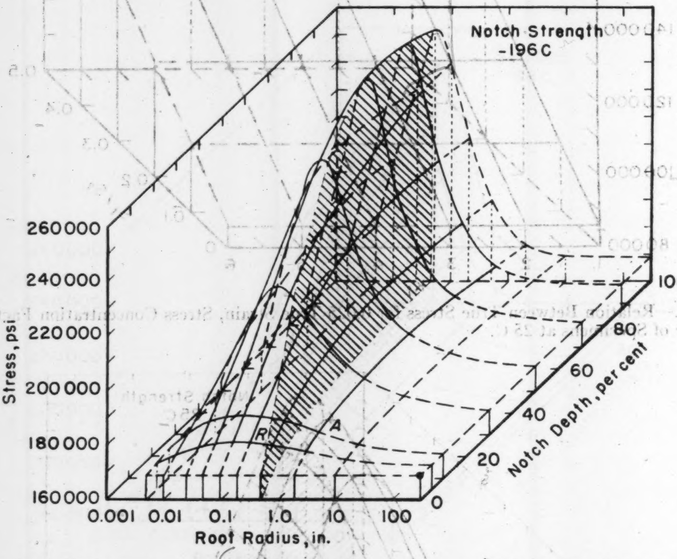


FIG. 15.—Relation Between Notch Strength, Root Radius, and Notch Depth of Specimens at −196 C.

Notch Strength.—The notch strength (Table I) of the specimens, in general, tended to increase with increase in notch depth or decrease in root radius and temperature. These relationships are illustrated by the notch strength surfaces presented in Figs. 14 and 15 for specimens tested at 25 and −196 C, respectively. Similar notch strength surfaces (not shown) were obtained for specimens tested at 100 and −78 C. The notch strength values over certain ranges of small root radii, however, decreased with decrease in the radius. Moreover, for the deep-notched specimens the upper limit of these ranges of root radii extended to larger radii with decrease in temperature. This feature can be attributed directly to the fracture of these specimens at true strains considerably below those corresponding to normal maximum load conditions. It should be noted that the notch strength (Figs. 14 and 15) of the specimens in which the fracture was initiated at or near the axis increased with decrease in the root radius. Conversely, the notch strength, especially at −78 and −196 C, of the specimens in which the fracture was initiated at or near the root of the notch, in general either increased very little or decreased with decrease in the root radius.

The notch strength of the titanium

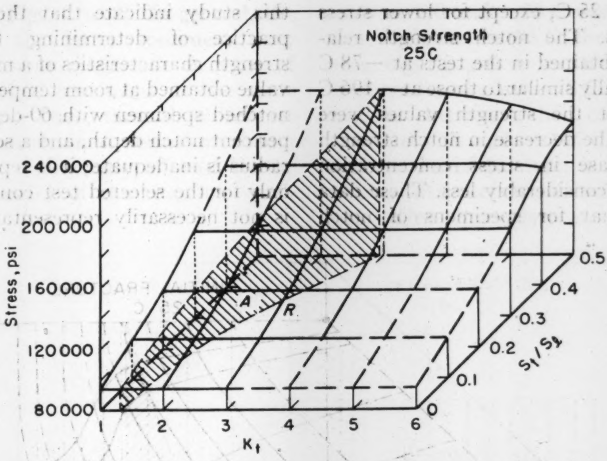


FIG. 16.—Relation Between Notch Strength, Stress Concentration Factor, and Triaxiality of Specimens at 25 C.

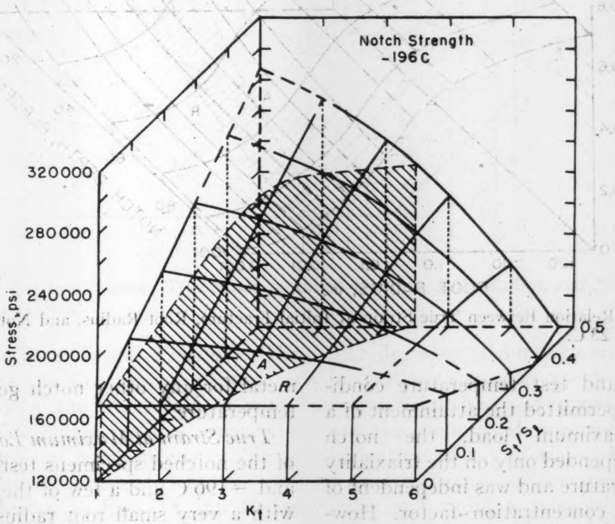


FIG. 17.—Relation Between Notch Strength, Stress Concentration Factor, and Triaxiality of Specimens at −196 C.

specimens depended primarily on the triaxiality and only indirectly on the stress concentration factor. These relationships are illustrated by the strength surfaces presented in Figs. 16 and 17 for the tests at 25 and −196 C, respectively. The corresponding strength relationships at 100 C (not shown) were very similar to those at 25 C, except for lower stress magnitudes. The notch strength relationships obtained in the tests at −78 C were generally similar to those at −196 C except that the strength values were lower and the decrease in notch strength with increase in stress concentration factor was considerably less. These data indicate that for specimens of notch geometry and test temperature conditions that permitted the attainment of a normal maximum load, the notch strength depended only on the triaxiality and temperature and was independent of the stress concentration factor. However, for specimens that did not retain sufficient ductility to reach a normal maximum load condition (Fig. 17 and upper right portion of Fig. 16) the notch strength tended to decrease with increase in stress concentration factor.

The notch strength data obtained in this study indicate that the prevalent practice of determining the notch strength characteristics of a metal by the value obtained at room temperature on a notched specimen with 60-deg angle, 50 per cent notch depth, and a selected root radius is inadequate; it is representative only for the selected test condition, and is not necessarily representative of the metal for any other notch geometry or temperature.

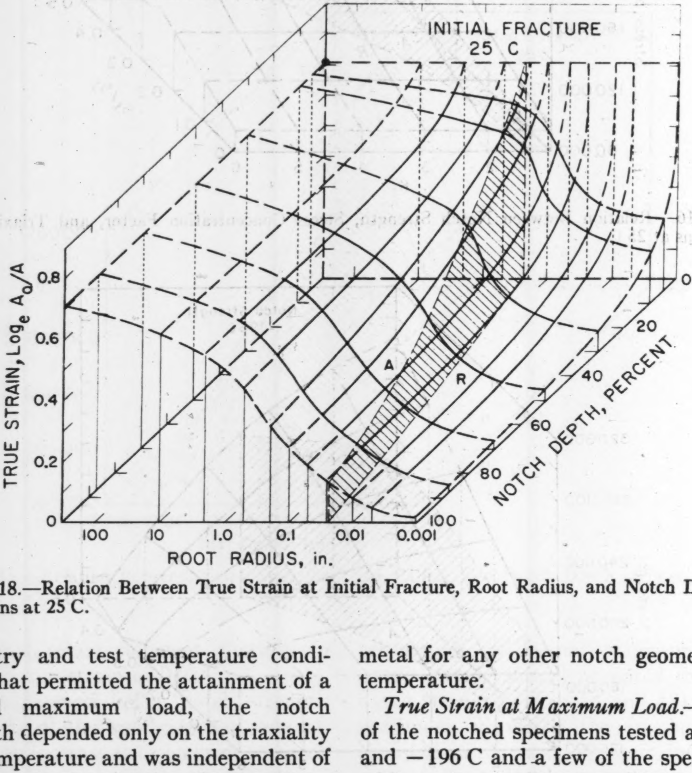


FIG. 18.—Relation Between True Strain at Initial Fracture, Root Radius, and Notch Depth of Specimens at 25 C.

True Strain at Maximum Load.—Most of the notched specimens tested at −78 and −196 C and a few of the specimens with a very small root radius tested at 25 C did not retain sufficient ductility to attain a normal maximum load condition (Table I, and Figs. 3, 4, 5, 7, and 8). Moreover, there was some scatter in the observed values of true strain at maximum load of the notched specimens that did reach a normal maximum load condition (Figs. 2 to 8). In general, the strains of the latter specimens were about the same magnitude as those of the unnotched specimens at the same temperature. Thus, these data indicate that the true strain at normal maximum load is not significantly affected by the notch geometry of the titanium specimens.

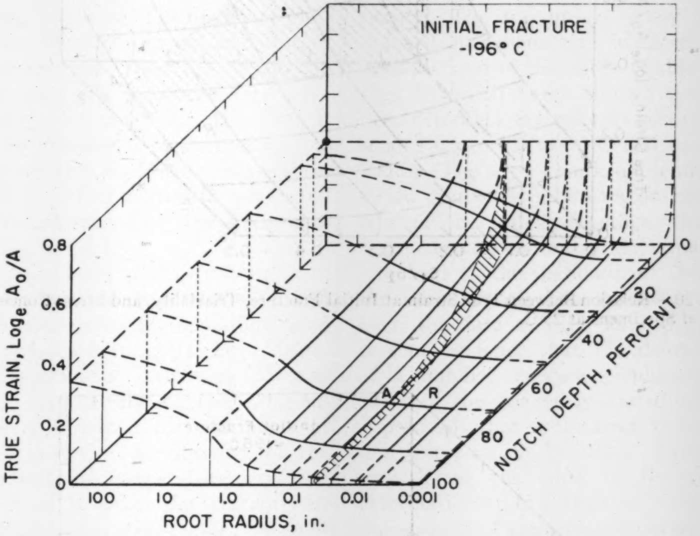


FIG. 19.—Relation Between True Strain at Initial Fracture, Root Radius, and Notch Depth of Specimens at −196 C.

True Strain at Initial Fracture.—The ductility of the titanium specimens, as determined by the true strain at initial fracture,[7] depended greatly upon the temperature and notch geometry. These relationships are illustrated by the surfaces delineated in Figs. 18 and 19 for specimens tested at 25 and −196 C, respectively. The corresponding ductility surfaces (not shown) obtained for specimens at 100 and −78 C were similar to those at 25 and −196 C; the true strains were slightly greater at 100 C than at 25 C, and those at −78 C were intermediate between the corresponding values at 25 and −196 C. The true strain at initial fracture, in general, decreased with decrease in temperature or root radius, or increase in notch depth. The decrease in ductility with decrease in temperature was much more pronounced for the notched specimens than for the unnotched specimens; this is indicative of a material that is notch-sensitive. Except for the specimens with large root radii, a relatively large decrease in the true strain at initial fracture was observed as the notch depth was increased to about 10 per cent at −196 C (Fig. 19) and to about 30 per cent at the other temperatures (Fig. 18 and Table I). The decrease in the ductility with further increase in the notch depth was usually

---

[7] The true strain and true stress values at initial fracture are determined from the simultaneous load and diameter measurements taken immediately preceding the very rapid propagation of the fracture crack across the specimen.

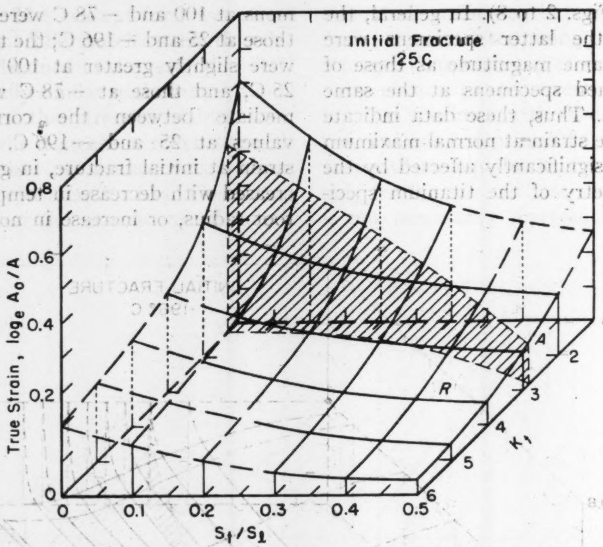


FIG. 20.—Relation Between True Strain at Initial Fracture, Triaxiality, and Stress Concentration Factor of Specimens at 25 C.

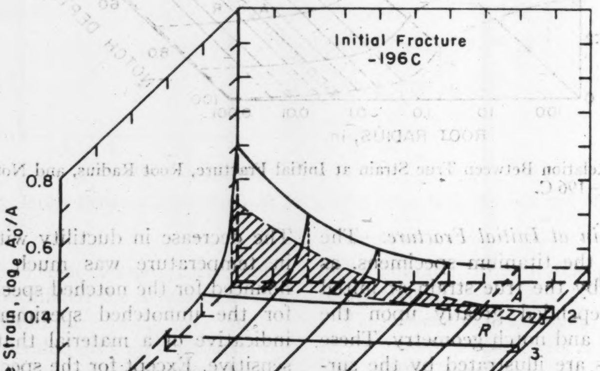


FIG. 21.—Relation Between True Strain at Initial Fracture, Triaxiality, and Stress Concentration Factor of Specimens at −196 C.

less pronounced. The dependence of ductility on root radius was also very pronounced. This is illustrated in Figs. 18 and 19 by the sharp drop in the ductility surfaces as the root radius was decreased through a range of values which varied some with the notch depth. In general, this sharp decrease in ductility with decrease in root radius was associated with the larger radii as the notch depth was increased. The boundary indicating the transition in the initiation of the fracture crack from the axis to the root of the notch is located near these same ranges of root radii. However, it should be noted that this transition occurs at smaller radii with increase in notch depth. This feature is associated with induced triaxiality and stress concentration factor.

The dependence of the true strain at initial fracture on the triaxiality and stress concentration factor is illustrated by the surfaces delineated in Figs. 20 and 21 for specimens tested at 25 and −196 C, respectively. Similar relationships were obtained for specimens at 100 and −78 C. The ductility of the specimens at each temperature decreased with increase in either the triaxiality or the stress concentration factor; the combined effect of these two factors on the ductility was quite pronounced, especially for the lower ranges of values of these two factors (Fig. 20). Moreover, it became even more pronounced with decrease in temperature (Fig. 21).

The initial stress concentration and accompanying work hardening and strain gradients developed during the deformation of the specimen tend to initiate the fracture crack at or near the root of the notch. Conversely, the induced triaxial stresses tend to initiate the fracture crack at or near the axis. Thus, the transition boundaries (hatched sections in the figures) may be considered as representing a balance between the effects of the above two factors on the location of the initiation of the fracture.

The ductility and fracture initiation data obtained in this investigation provide direct evidence that deformation, even to relatively large strains, does not completely remove the embrittling effects of the initial stress concentration and associated work hardening and strain gradients on tensile behavior of the titanium. The fracture of many of the notched specimens was initiated at or near the root of the notch (Table I and Figs. 18 to 21) even though they had been deformed to large true strains (true strains up to 0.5 at 25 C and to 0.6 at 100 C). The severe work hardening and strain gradients in the region near the root of the notch, together with any retained stress concentration, apparently remained as the predominant factor in the initiation of these fractures. However, the data in Figs. 20 and 21 also present evidence that the deformation of a notched specimen enhances the effect of the triaxiality and diminishes appreciably that of the stress concentration factor on the initiation of the fracture. Thus, the effect of the greater deformation at 25 C than at −196 C on the position of the transition boundaries (hatched sections) is appreciable; that is, the boundary at 25 C (Fig. 20) is located at higher K_t values than the boundary at −196 C (Fig. 21).

True Stress at Initial Fracture.—No simple relationships were observed between the true stress at initial fracture (hereafter designated as fracture stress) and the notch geometry. This strength index depends on the variations of both the ductility and resistance to flow with the notch geometry. The observed variation of the fracture stress with the notch depth and root radius is illustrated by the surfaces delineated in Figs. 22 and 23 for specimens fractured at 25 and −196 C, respectively. (It should be

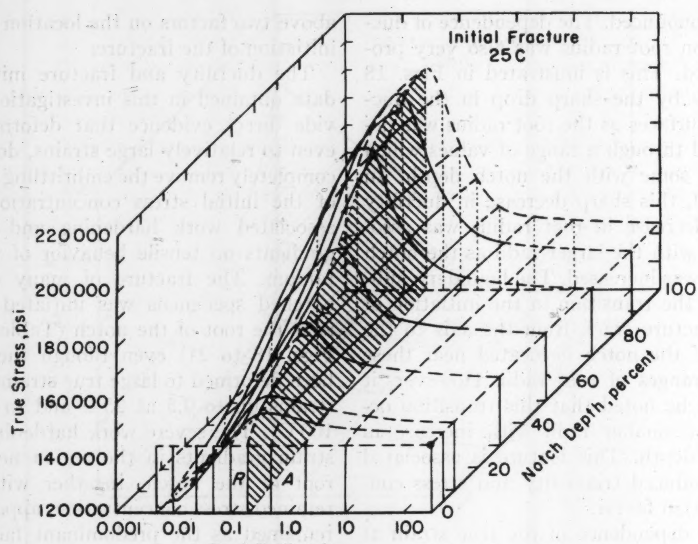


FIG. 22.—Relation Between True Stress at Initial Fracture, Root Radius, and Notch Depth of Specimens at 25 C.

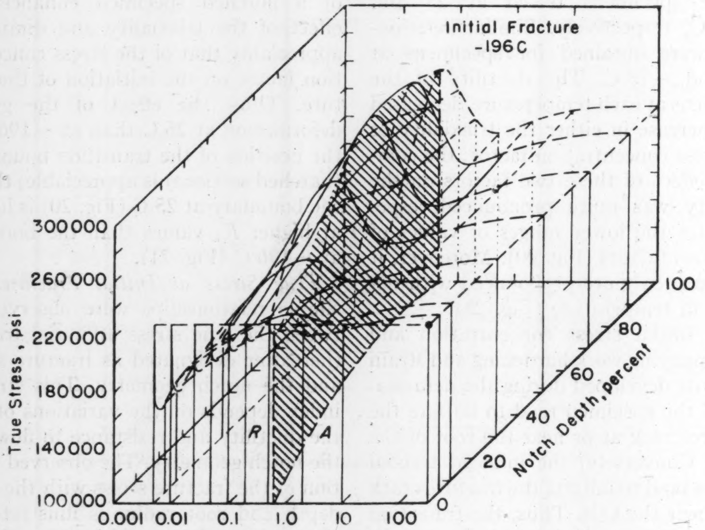


FIG. 23.—Relation Between True Stress at Initial Fracture, Root Radius, and Notch Depth of Specimens at −196 C.

noted that the ordinate scales are different in these figures.) Similar surfaces were obtained for specimens fractured at 100 and −78 C for this strength index; the stress values at 100 and −78 C were, in general, approximately 20 per cent lower and higher, respectively, than those for corresponding specimens at 25 C.

The fracture stress, as shown by the curves in Figs. 2 to 8, tends to (1) increase with increase in the resistance to flow and (2) decrease with decrease in the ductility of the specimens. Thus, this strength index depends greatly upon which of the above two factors is predominant for the selected notch geometry and temperature. The fracture stresses at 100, 25, and −78 C (Table I and Fig. 22) of the specimens in which the fracture crack was initiated at or near the axis depended primarily upon the resistance to flow and secondarily upon the ductility of the specimens; it increased with increase in notch depth, or decrease in root radius or temperature. Conversely, for the specimens at 100, 25, −78, and −196 C in which the fracture crack was initiated at or near the root of the notch (Table I and Figs. 22 and 23), the fracture stress depended primarily on the ductility and secondarily on the resistance to flow of the specimens; it decreased rapidly with the decrease in ductility accompanying the decrease in root radius. This latter feature was very prominent for the deep-notched specimens at −196 C.

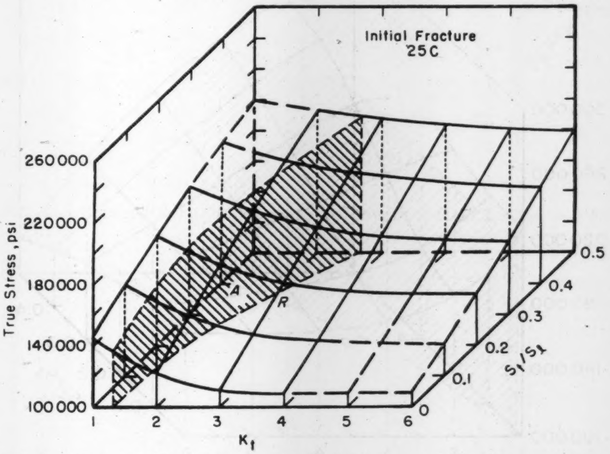


FIG. 24.—Relation Between True Stress at Initial Fracture, Stress Concentration Factor, and Triaxiality of Specimens at 25 C.

The fracture surface (Fig. 23) obtained for the specimens at −196 C exhibits a feature that was not observed at the other temperatures. This is the general decrease in the fracture stress of specimens of large root radii with decrease in the radius to 0.5 in. At this temperature, the decrease in fracture stress over this range of radii can be attributed to the predominance of the ductility factor on the fracture stress; the ductility of these specimens, especially the deep-notched specimens (Fig. 19), generally decreased rapidly with decrease in the root radius.

The relationships observed between the fracture stress and the triaxiality or the stress concentration factor of the notched specimens are illustrated by the surfaces delineated in Figs. 24 and 25 for the specimens fractured at 25 and -196 C, respectively. The corresponding fracture stress relationships for the tests at 100 and -78 C were similar to those at 25 C. These data present additional evidence that the fracture stress generally increases with increase in the triaxiality of the specimen and decreases with increase in the stress concentration factor. The decrease in the fracture stress with increase in the stress concentration factor can be attributed directly to the effect of stress concentrations, accompanying work hardening, and strain gradients in limiting the ductility of the specimens. (See discussion under True Strain at Initial Fracture, and compare Figs. 24 and 25 with Figs. 20 and 21.)

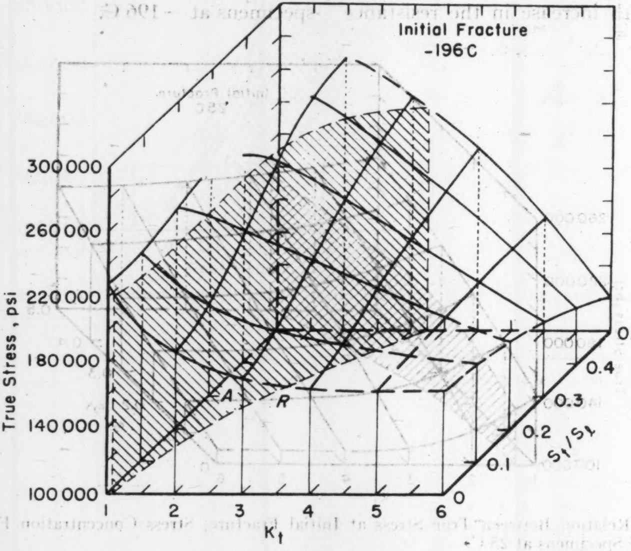


FIG. 25.—Relation Between True Stress at Initial Fracture, Stress Concentration Factor, and Triaxiality of Specimens at -196 C.

Energy of Deformation.—The true stress - true strain data obtained in this investigation provide a means of determining an approximate measure of the relative energy of deformation[8] of the notched specimens. These energy values are given in Table I; the relative energy values for specimens at 100, -78, or -196 C may be obtained in terms of the energy of deformation of unnotched specimens at 25 C by multiplying the corresponding series of values (column 15, Table I) by 0.88, 0.93, and 0.75, respectively. (These multiplication factors are the ratios of the areas under the true stress - true strain curves of the unnotched specimens at 100, -78, and

---

[8] Relative energy of deformation at a selected temperature is determined as the ratios of the area under the true stress - true strain curves of the notched and unnotched specimens; these ratios are a comparison of the maximum strain energy densities of the specimens, and are not ratios of the total strain energy of deformation.

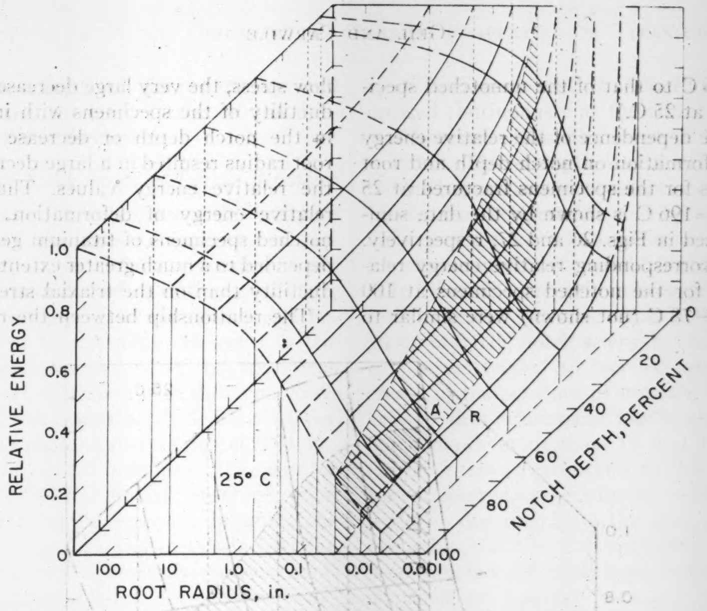


FIG. 26.—Relation Between Relative Energy of Deformation, Root Radius, and Notch Depth of Specimens at 25 C.

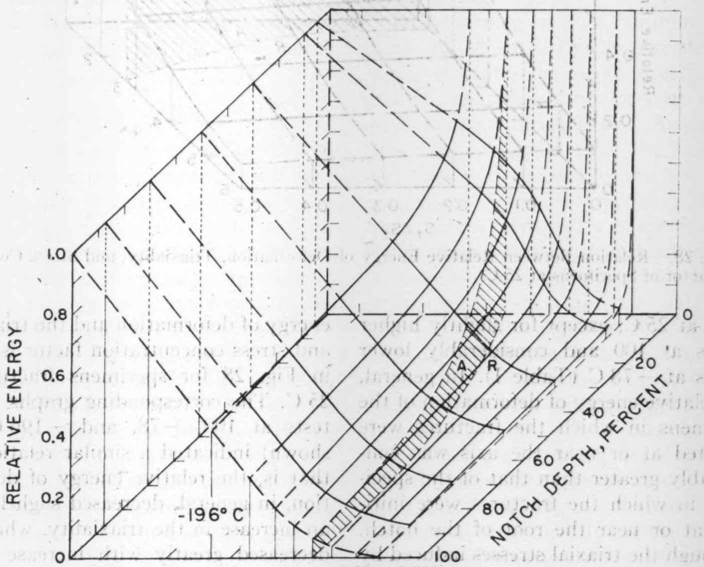


FIG. 27.—Relation Between Relative Energy of Deformation, Root Radius, and Notch Depth of Specimens at −196 C.

−196 C to that of the unnotched specimens at 25 C.)

The dependence of the relative energy of deformation on notch depth and root radius for the specimens fractured at 25 and −196 C is shown by the data summarized in Figs. 26 and 27, respectively. The corresponding relative energy relations for the notched specimens at 100 and −78 C (not shown) were similar to those at 25 C, except for slightly higher values at 100 and considerably lower values at −78 C (Table I). In general, the relative energy of deformation of the specimens in which the fractures were initiated at or near the axis was considerably greater than that of the specimens in which the fractures were initiated at or near the root of the notch. Although the triaxial stresses induced by the notch tended to increase the energy of deformation due to the increase in flow stress, the very large decrease in the ductility of the specimens with increase in the notch depth or decrease in the root radius resulted in a large decrease in the relative energy values. Thus, the relative energy of deformation of the notched specimens of titanium generally depended to a much greater extent on the ductility than on the triaxial stresses.

The relationship between the relative energy of deformation and the triaxiality and stress concentration factor is shown in Fig. 28 for specimens fractured at 25 C. The corresponding graphs for the tests at 100, −78, and −196 C (not shown) indicated a similar relationship; that is, the relative energy of deformation, in general, decreased slightly with an increase in the triaxiality, whereas it decreased greatly with increase in the stress concentration factor. These features can be attributed mainly to the

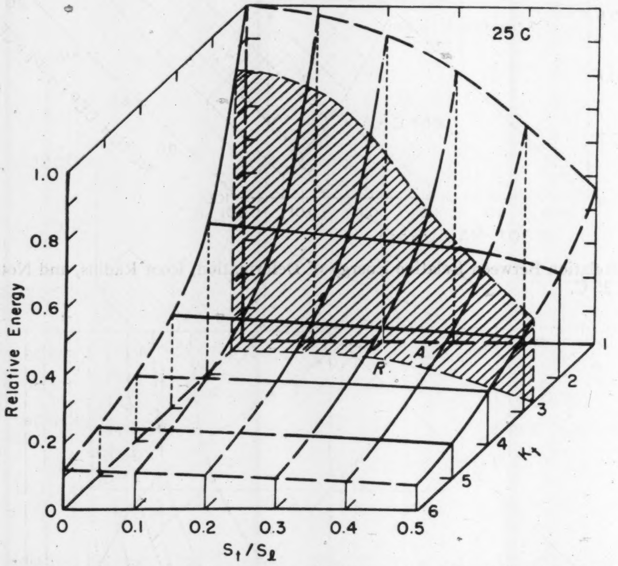


FIG. 28.—Relation Between Relative Energy of Deformation, Triaxiality, and Stress Concentration Factor of Specimens at 25 C.

adverse effects of the triaxiality and stress concentration factor on the ductility of the specimens.

Effect of Notch Angle on Tensile Behavior:

It was not feasible, due to the extensive scope of the present experimental investigation, to include a detailed study of the combined effect of the notch angle and low temperatures on the tensile behavior of the titanium. However, a few tests were made at 25 C on specimens that were machined with different notch angles, a constant notch depth of 50 per cent, a constant root radius of 0.01 in., and a constant minimum diameter of 0.350 in. The results observed were similar to those reported in numerous publications for other metals in that the strength indices generally increased and the ductility decreased greatly with decrease in the notch angle and the accompanying increase in triaxiality and stress concentration factor. The relative energy of deformation of these specimens also decreased with decrease in the notch angle.

SUMMARY AND CONCLUSIONS

A study was made of the combined effects of low temperatures and the geometry of a notch on the tensile behavior of specimens of annealed commercially pure titanium. The tensile specimens were of circular cross-section and the notches were machined to selected depths (constant diameter of 0.350 in. under the notch) and root radii. The notch depth varied from 5 to 87 per cent, the root radius from 0.005 to 2.0 in., and test temperature from 100 to −196 C. True stress - true strain data were obtained from simultaneous load and minimum diameter measurements made throughout the duration of each tension test.

The resistance to flow and other strength indices of the unnotched specimens increased and the ductility decreased continuously as the temperature was lowered within the range of 100 to −196 C. The strength and ductility of notched specimens were affected greatly by the test temperature and by the geometry of the notch and the accompanying stress concentration and triaxiality.

In general, strength indices such as resistance to flow, true stress at a true strain of 0.025, notch strength, and fracture stress tended to increase with decrease in temperature or increase in triaxiality that accompanied an increase in notch depth or decrease in root radius. However, many exceptions to this trend were observed, especially at −78 and −196 C, due mainly to the very low or negligible ductility of specimens with deep notches or small root radii. The relationships between strength indices and these variables are shown in representative graphs and discussed in some detail in the text.

The ductility of the titanium specimens, as measured by the true strain at initial fracture, decreased greatly with decrease in temperature or root radius, or increase in notch depth. Analysis of these data showed that the ductility decreased with increase in the stress concentration factor or triaxiality of the specimens. The ductility of specimens in which the fracture crack was initiated at or near the root of the notch by the stress concentration and associated factors was usually less than that of specimens in which the fracture crack was initiated at or near the axis by the triaxial stresses; the embrittlement of the titanium by the stress concentration and accompanying work hardening and strain gradients near the root of the notch was generally greater than the embrittlement due directly to the triaxial stresses. Deformation of specimens, even to relatively large strains, reduced somewhat but did not completely remove the embrittling effect of the stress concentration at the root of the notch.

The relative energy of deformation of the notched specimens depended primarily on the ductility and secondarily on the triaxiality. Although the resistance to flow increased with increase in triaxiality of the specimens, the relative energy of deformation generally decreased with increase in triaxiality or stress concentration factor due to the very large decrease in the ductility of the specimens.

The results of this study clearly demonstrate that the tensile behavior at low temperatures of specimens with different notch geometries cannot, in general, be predicted from the observed tensile behavior at a single temperature of specimens with a fixed notch geometry. Furthermore, the results emphasize the importance of the general requirement that in designing for low-temperature service of titanium or other metals and alloys special care should be taken to eliminate notches wherever possible. However, if notches are unavoidable, then attempts should be made to mitigate their embrittling effects by designing the notch with the largest permissible root radius.

REFERENCES

(1) J. W. Spretnak, M. G. Fontana, and H. E. Brooks, "Notched and Unnotched Tensile and Fatigue Properties of Ten Engineering Alloys at 25°C and −196°C," *Transactions*, Am. Soc. Metals, Vol. 43, p. 547 (1951).

(2) S. M. Bishop, J. W. Spretnak, and M. G. Fontana, "Mechanical Properties Including Fatigue of Titanium Base Alloys RC 130B and Ti 75A at Very Low Temperatures," *Transactions*, Am. Soc. Metals, Vol. 45, p. 993 (1953).

(3) H. R. Ogden, F. C. Holden, and R. I. Jaffee, "The Effect of Microstructure on the Mechanical Properties of Titanium Alloys—Properties of Titanium Alloys as Affected by Temperature and Notches," Fifth Interim Report (Contract No. DA-33-019 ORD-1397), Battelle Memorial Inst. to Watertown Arsenal (Jan. 3, 1955).

(4) E. P. Klier and N. J. Feola, "Notch Tensile Properties of Selected Titanium Alloys," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 209, p. 1271 (1957).

(5) R. I. Jaffee, G. A. Lenning, and C. M. Craighead, "Effect of Testing Variables on the Hydrogen Embrittlement of Titanium and a Ti - 8 Pct Mn Alloy," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 206, p. 907 (1956).

(6) F. R. Brotzen, E. L. Harmon, Jr., and A. R. Troiano, "How Notch Sensitive Are Titanium Alloys?" *Iron Age*, Vol. 174, p. 52, Dec. 30, 1954.

(7) E. L. Harmon, J. Kozol, and A. R. Troiano, "Mechanical Properties Correlated with Transformation Characteristics of Titanium-Vanadium Alloys," *Transactions*, Am. Soc. Metals, Vol. 50, p. 418 (1958).

(8) G. W. Geil and N. L. Carwile, "Some Effects of Low Temperatures and Notch Depth on the Mechanical Behavior of an Annealed Commercially Pure Titanium," *Journal of Research*, National Bureau of Standards, Vol. 59, p. 215 (1957).

(9) G. W. Geil and N. L. Carwile, "A Reduction-of-Area Gage for Use at Low Temperatures," *Journal of Research*, National Bureau of Standards, Vol. 43, p. 527 (1949); also ASTM BULLETIN, No. 163, Jan., 1950, p. 75 (TP25).

(10) G. W. Geil and N. L. Carwile, "Tensile Properties of Ingot Iron at Low Temperatures," *Journal of Research*, National Bureau of Standards, Vol. 45, p. 129 (1950).

(11) G. W. Geil and N. L. Carwile, "Tensile Properties of Copper, Nickel and Some Copper-Nickel Alloys at Low Temperatures," *National Bureau of Standards Circular 520*, p. 67 (1952).

(12) R. E. Peterson, "Stress Concentration Design Factors," John Wiley & Sons, Inc., New York, N. Y. (1953).

(13) G. Sachs and J. D. Lubahn, "The Effect of Triaxiality on the Technical Cohesive Strength of Steels," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 67, p. A240 (1945).

DISCUSSION

Mr. R. I. Jaffee.[1]—The authors are discussing unalloyed titanium metal showing how low temperature increased the notch sensitivity of this material.

I would like to point out one additional embrittlement range which can be detected if notch sensitivity tests are conducted over a range of temperatures. We found in three materials, Ti-6Al-4V, Ti-140A, and Ti-5Al-2.5Sn, that in the temperature range of about 0 to −40 C, there is embrittlement due to hydrogen which would not have shown up in unnotched tests. This occurred even when the hydrogen content was well below 100 ppm. Presumably, the presence of the notch decreased the amount of hydrogen needed to show embrittlement in room-temperature, notch-rupture tests.

Mr. G. W. Geil (*author*).—The hydrogen content of the titanium was 120 ppm.

Mr. Herbert T. Corten.[2]—This material, like steel, is quite sensitive to the rate of straining. I assume that all of the specimens were tested at the same loading rate, which means that for the sharper notches, the material where the cracks initiated was subjected to a higher rate of straining. Thus some effect of rate of straining was introduced as well as the variables that were mentioned in the paper. Would the author comment upon this?

Mr. Geil.—The specimens were tested at approximately the same loading rate up to the initial yielding. However, for the deformation beyond initial yielding the deformation rate was maintained at about 0.5 to 1.0 per cent contraction in area per minute. Thus, although the original minimum cross-sectional area of the notched specimens was constant (0.350 in.) and the average rate of straining (contraction in area at the minimum cross-section) of the different notched specimens did not vary significantly, it is reasonable to assume that the rate of straining of the metal in the region adjacent to the root of the notch varied some with the sharpness of the notch. However, the authors believe that the effect of this rate factor on the initiation of the fracture was relatively small.

Mr. Dale Dickinson.[3]—Do you know the oxygen content of the titanium?

Mr. Geil.—The oxygen content is reported in the paper; it was 0.21 per cent.

Mr. Dickinson.—Then, this is relatively impure titanium representative of commercial practice.

Mr. Geil.—This material was a commercial purity titanium that corresponds to the trade composition, designated as Ti-75A. Thus it was not a high-purity titanium.

Mr. Dickinson.—If I am not mistaken oxygen has a deleterious effect on the transition temperature behavior of titanium and its alloys.

Mr. Geil.—We have tested a titanium of higher purity in which the nitrogen and carbon contents were one half and the oxygen content was one third as great as those in this material and it had much higher ductility, especially at low temperatures. The transition temperature ranges as determined on Charpy V-notch specimens of these two samples were nearly the same; the higher purity sample had a slightly higher transition temperature, as determined by the temperature corresponding to the steepest portion of the energy absorbed *versus* temperature curve. However, the values of the energy absorbed in the impact tests, especially at temperatures below the transition temperatures, were greater for the higher purity titanium.

---

[1] Battelle Memorial Institute, Columbus, Ohio.

[2] Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[3] Manager, Fundamental Research, Mallory-Sharon Metals Corp., Niles, Ohio.

Mr. John G. Sessler.[4]—This work was done on a rather ductile material, or low-strength titanium material. I was wondering if you people had contemplated working with some of the heat-treated titanium alloys, such as Ti-6Al-4V or Ti-4Al-3Mo-1V for the same type of study.

Mr. Geil.—No, we have not and it is not included in our present plans. However, some similar work was carried out on an annealed, 4 per cent aluminum - 4 per cent manganese alloy (but not in any heat-treated conditions), and the results were similar to those reported in this paper except for lower ductility as a whole.

---

[4] Project Engineer, Syracuse University Research Inst., Syracuse, N. Y.

STRESS-STRAIN PROPERTIES OF SELECTED TITANIUM METALS*

By E. P. Klier[1] and C. P. Gazzara[2]

Synopsis

The plastic flow properties of three titanium metals as function of four strain rates and five temperature levels were studied. The stress-strain relationships were analyzed on the basis of the power law: $\sigma = \sigma_0\delta^n$ and were found to be characterized by three regions: (1) that dominated by the yield phenomenon associated with the strain hardening exponent n_1 and the strength coefficient $\sigma_{0,1}$; (2) that related to a strain hardening phenomenon describable by a second strain hardening exponent n_2 and strength coefficient $\sigma_{0,2}$; and (3) an intermediate strain region, represented by an overlapping of the two stress-strain curves relative to regions (1) and (2) promoting a maximum intermediate stress.

For the past half century, a suitable representation of the flow of a metal through work hardening has been the power law representation advanced by Ludwik (**1**).[3] Hollomon (**2**) has proposed that Ludwik's equation is sufficiently accurate when modified to disregard the elastic strain interval, and several reports have been offered to support this suggestion (**3**,**4**). The agreement between the equation and experimental data is seldom exact and it has been proposed on occasion that it is not adequate (**5**,**6**). However, despite its inadequacies the simplified representation does faithfully agree with the experimental data over a very large strain interval, and the constants in the expression, that is, n and σ_0 in $\sigma = \sigma_0\delta^n$, have a direct physical and mathematical significance. If the power law holds, for example, it can be shown that $n = \delta_{\text{max load}}$. It should, therefore, be evident from inspection of the stress-strain curve. Recently it has been shown, however, that for titanium (**7**) n cannot, in general, be determined from inspection of the stress-strain curve in a manner suggesting straightforward application of the expression $\sigma = \sigma_0\delta^n$. The present work, consequently, was completed in order to establish more satisfactorily the flow properties of titanium materials.

Experimental Procedure

Titanium Materials:

Titanium ingots of approximately 7 lb were consumable electrode melted. The titanium-aluminum alloys were prepared by the addition of pure aluminum, while the titanium-manganese alloys were prepared by the addition of electrolytic

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Research Professor of Metallurgy, The Catholic University of America, Washington, D. C.

[2] Watertown Arsenal Laboratories, Watertown Arsenal, Watertown, Mass.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—COMPOSITIONS OF ALLOYS DISCUSSED.

Material	Lot Number	Carbon, per cent	Nitrogen, per cent	Oxygen, per cent	Hydrogen, per cent
Unalloyed titanium	52	0.02	0.02	0.063	0.005
	55	0.04 to 0.05	0.02	0.073	0.0034
	AA	0.05 to 0.06	0.02	0.081	0.0058
5 Al titanium	16	0.03	0.03	0.066	0.016
6.5 Mn titanium	29	0.05	0.09	0.159	0.012
	31	0.10 to 0.11	0.04	0.153	0.0088

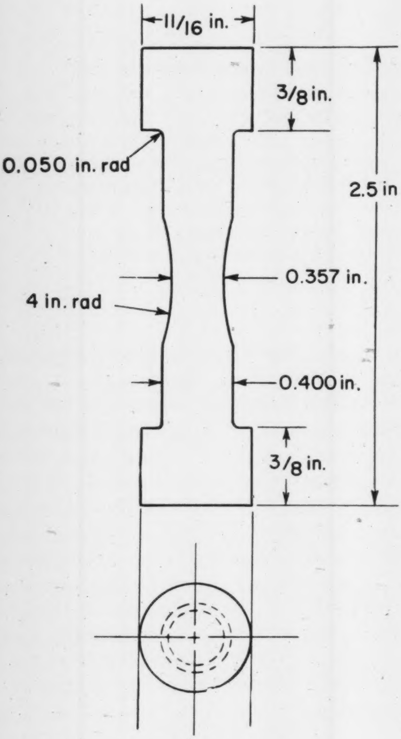


FIG. 1.—The Tension Specimen.

manganese. In all about 27 ingots were melted and prepared for testing. In the present paper only six of these materials are discussed in detail, and the compositions of these six materials only are reported in Table I. The ingots were cleaned and forged to $\frac{3}{4}$-in. diameter rod which was then annealed as follows:

	Forged, deg Fahr	Annealing Treatment
Unalloyed titanium	1600	1460 F (1 hr), furnace cool
5 Al titanium	1950	1400 F (1 hr), furnace cool
6.5 Mn titanium	1650	1250 F (1 hr), furnace cool, 1000 F (8 hr), furnace cool

Tension Test Specimens:

Tension specimens of the dimensions shown in Fig. 1 were used. Since diameter measurements through the test at all strain rates were required, the contoured specimen was needed to localize maximum strain to allow satisfactory placement of the diameter gage. At the start of testing, the autographic diameter gage was located at the minimum diameter and was retained automatically at this location throughout the test.

Test Conditions:

Specimens were tested at 70, 0, −100, −200, and −320 F; and at $\dot{\delta}_1 = 2$ to 3 min^{-1}, $\dot{\delta}_2 = 0.5$ min^{-1}, $\dot{\delta}_3 = 0.07$ min^{-1}, and $\dot{\delta}_4 = 0.003$ to 0.004 min^{-1}. Load-diameter curves were plotted for each test condition. The diameter gage was in accordance with Geil and Carwile (8) but modified to allow the load-diameter curve to be autographically recorded.

Constant Strain Rate:

$$\delta = \text{natural strain} = ln \frac{D_0^2}{D_i^2}$$

$$\frac{d\delta}{dt} = \frac{-2}{D_i}\frac{dD_i}{dt}$$

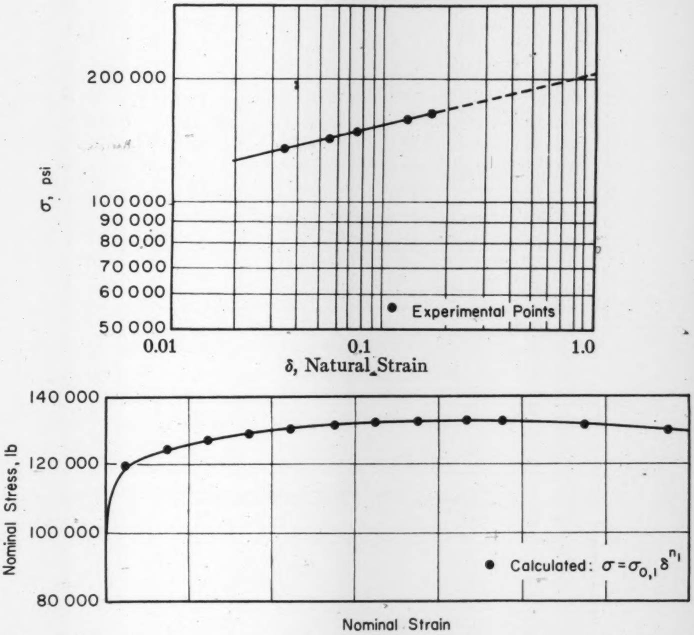


FIG. 2.—Stress-Strain Curves for 6.5 Mn Titanium. Test temperature, 70 F; strain rate 3.

where:

D_0 = original diameter,
D_i = instantaneous diameter, and
$dD/dt = \dot{\delta}$ = natural strain rate.

Through each test:

$$\frac{dD_i}{dt} = \text{constant, or}$$

$$\dot{\delta} = \frac{K}{D_i}; \quad K = \text{constant}$$

The strain rate thus varied as the diameter during the test. The maximum departure from constant strain rate due to the D_i term is approximately 50 per cent. Since $\dot{\delta}_1$, $\dot{\delta}_2$, $\dot{\delta}_3$, and $\dot{\delta}_4$ differ progressively by about a factor of 10, the relative error to maximum load is 5 per cent, for the entire test 10 per cent.

Data Processing:

The load-diameter curves were transferred to $ln\ \sigma$ - $ln\ \delta$ coordinates and the n and σ_0 values were measured. Elongation measurements were also made through each test and these data were used to give an adjusted strain measurement for the yield strength determination.

EXPERIMENTAL RESULTS

Characteristic Stress-Strain Curves:

On *ln-ln* coordinates the σ and δ in $\sigma = \sigma_0\delta^n$ form a linear relationship. An example of this behavior is given in Fig. 2 for a 6.5 Mn titanium alloy. Frequently, however, the experimental *ln* σ - *ln* δ curve formed two branches, three forms of which were observed. In Fig. 3(*a*), for example, the experimental points lie for practical purposes on the two straight line curves. These points generate the load - nominal strain curve in Fig. 3(*b*). The experimental curve in Fig. 3(*b*) corresponds to the envelope of the two calculated curves, which intersect smoothly in nominal stress-strain coordinates despite the discontinuity in the curve in *ln-ln* coordinates.

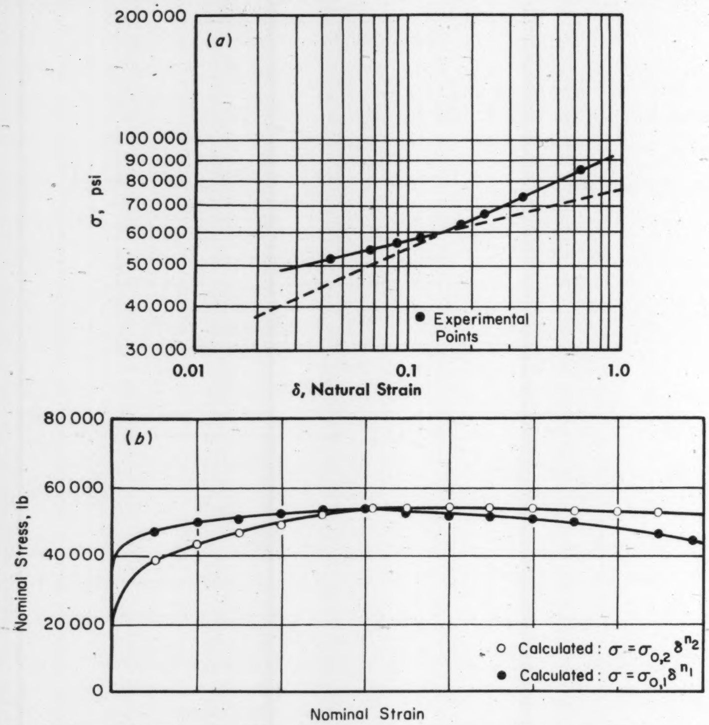


FIG. 3.—Stress-Strain Curves for Unalloyed Titanium. Test temperature, 70 F; strain rate 4.

When the experimental points in *ln-ln* coordinates pass smoothly from one straight line curve to the other, two possible nominal stress-strain curves arise. For the first, the first straight line curve lies at relatively low stresses so that the second strain interval dominates the maximum load strain (Fig. 4), while for the second, the first straight line curve lies at relatively high stresses and dominates the maximum load strain (Fig. 5). For both cases, an intermediate strain interval is closely approximated by the tangent line to the two calculated curves.

In the present experimental program more than 500 stress-strain curves were taken, and within the accuracy of the determination where it could be measured maximum load strain was found to agree adequately with either n_1 or n_2, or lay between these two values. In general, both temperature and strain rate changes affected the location of $\delta_{\text{max load}}$, with the characteristic effect of strain rate changes being illustrated in Fig. 6.

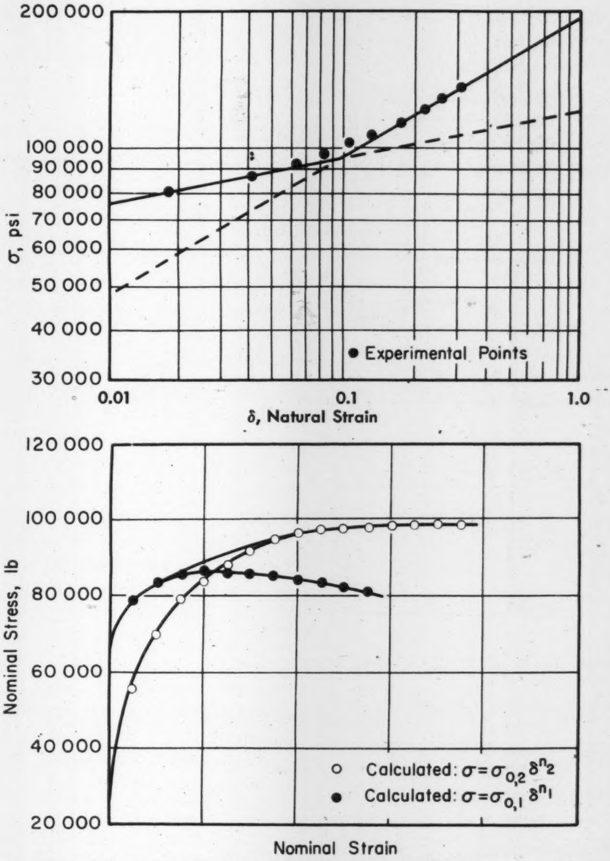


FIG. 4.—Stress-Strain Curves for Unalloyed Titanium. Test temperature, −200 F; strain rate 1.

At low strain rate for the alloy and testing temperature selected a stress-strain curve with double maxima is measured. With increasing strain rate, the early portion of the flow curve is raised and the intermediate stress minimum is eliminated. The flow curve as a whole is displaced to higher stresses. At the maximum strain rate the early portion of the flow curve is elevated due to the strain rate effect, but the test is now no longer isothermal; and due to the significant heating of the specimen through the test the stresses at high strains are relatively reduced (9). An increase in the strain rate displaces the low strain maximum to higher strains, while the second maximum is displaced to lower strains. Under

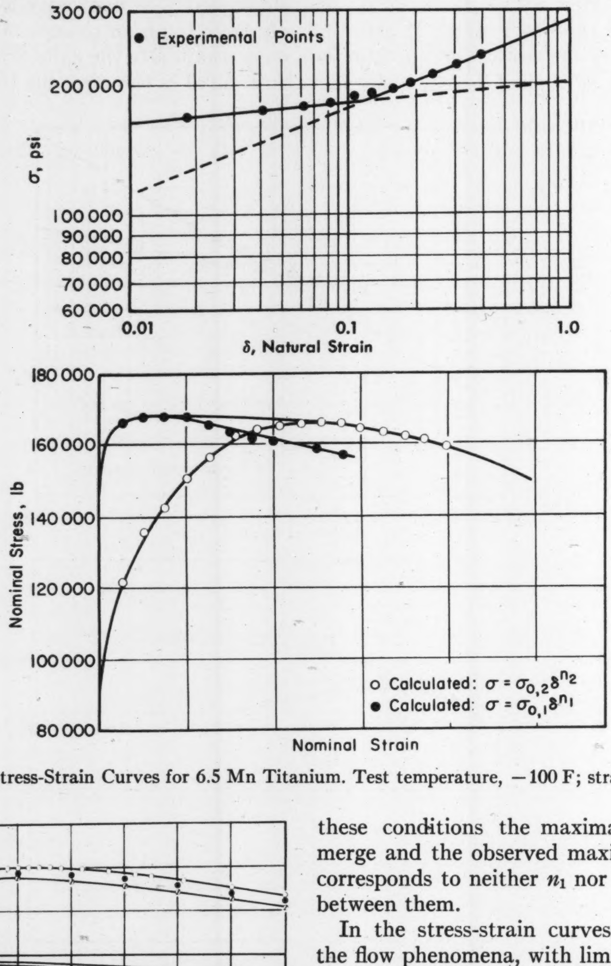
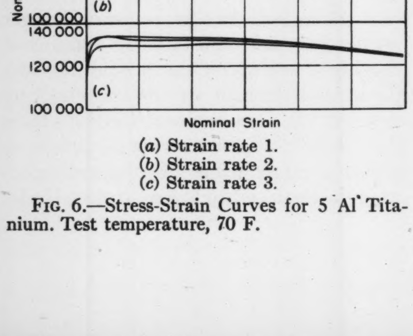


FIG. 5.—Stress-Strain Curves for 6.5 Mn Titanium. Test temperature, −100 F; strain rate 1.

these conditions the maxima tend to merge and the observed maximum then corresponds to neither n_1 nor n_2 but lies between them.

In the stress-strain curves presented the flow phenomena, with limited exceptions, have been too complex to represent by means of a single power law expression in the form of $\sigma = \sigma_0\delta^n$. However, by the use of two such expressions with the added provision for the construction of the tangent maximum envelope to the two consequent curves a faithful representation of the experimental flow curve becomes satisfactorily possible for strain rates usually employed in tension testing. At both higher and lower strain rates



(*a*) Strain rate 1.
(*b*) Strain rate 2.
(*c*) Strain rate 3.

FIG. 6.—Stress-Strain Curves for 5 Al Titanium. Test temperature, 70 F.

additional factors intrude and must be satisfactorily weighed to allow agreement between the experimental and calculated measurements. Thus at high strain rates there is a heating effect that varies through the test, while at low strain rates creep deformation becomes important. However, available evidence indicates that in the strain rate and test temperature intervals covered in this work the above two factors play a moderately small role and need not be explicitly examined for the purposes of the present investigation.

The stress-strain data for titanium are clearly more complicated than projected by simple agreement with $\sigma = \sigma_0 \delta^n$. However, it is desirable to establish, as the data allow, the significance of the two- or three-stage representation of the stress-strain relations for titanium. Preliminary consideration is directed to the value of n_1. For this purpose all values of n_1 for the respective alloys were assembled in frequency plots (Fig. 7). It is necessary to point out that while the nominal alloy for each frequency plot is fixed, the contamination level, which directly affects σ_0, is not constant through each plot. The comparisons made for n_1 cannot be made for σ_0.

The n_1 values were relatively unchanged with strain rate and temperature change, as well as with contamination change. The data assembled in Fig. 7 consequently are not differentiated with respect to temperature and strain rate.

The value for n_1 in unalloyed titanium has an average of 0.12, while in both alloys the average is about 0.07. In the unalloyed titanium the value of n_1 is, in general, significantly less than the instability strain. For the alloyed titanium, on the other hand, since the yield strength is relatively high compared to the tensile strength, the value n_1 may correspond to the instability strain and the latter consequently may be quite low.

The Stress-Strain Relationships in Unalloyed Titanium:

The three lots of unalloyed titanium were tested under the various conditions of temperature and strain rate. The average results for the three tests are presented in Figs. 8, 9, and 10.

The tensile and yield strengths increase with reduction in test temperature in full agreement with the data published elsewhere (10,11,12). As the strain rate is increased the strength also increases, but there is a slight trend toward the development of a minimum near the maximum strain rate in the tensile strength - strain rate curves. This has been reported elsewhere (9) as due to adiabatic heating in the specimen.

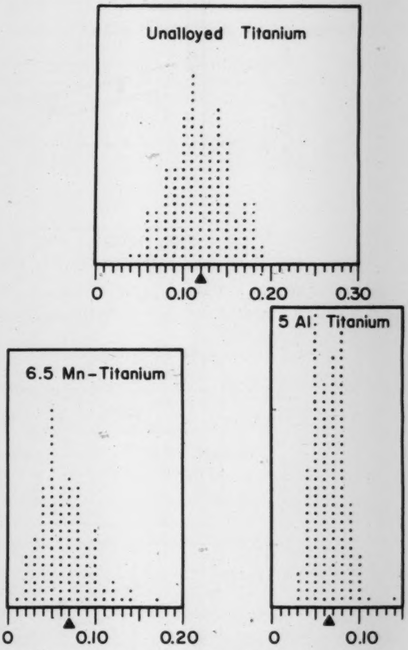


FIG. 7.—Frequency Distribution of the n_1 Values for the Indicated Materials.

The reduction in area is reduced as the

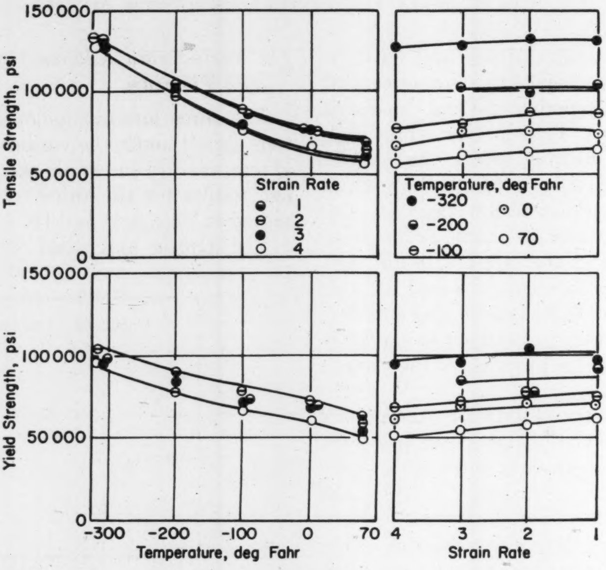


FIG. 8.—Strength Properties of Unalloyed Titanium as Function of Temperature and Strain Rate. (Values averaged for lots of metal 52, 55 and AA.)

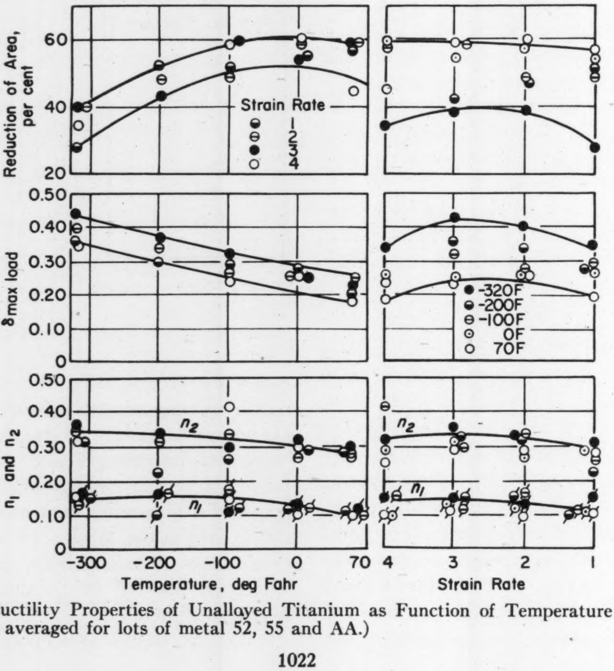


FIG. 9.—Ductility Properties of Unalloyed Titanium as Function of Temperature and Strain Rate. (Values averaged for lots of metal 52, 55 and AA.)

test temperature is lowered below −100 F, but is still 30 per cent or greater at −320 F. The reduction in area is slightly reduced as the strain rate is increased, but this trend is not strong except for the specimens tested at −320 F.

The strain at maximum load is strongly influenced by temperature change, being about doubled as the temperature is reduced from 70 to −320 F. At −320 F the $\delta_{\text{max load}}$ was of the same order of magnitude as the fracture strain so that it was frequently difficult to measure. However, an interesting trend is established for the $\delta_{\text{max load}}$ - temperature curve which, if confirmed at lower test temperatures, would signify that $\delta_{\text{max load}} \neq n$. It will be necessary to complete suitable tests on other more ductile materials at lower temperatures to determine if the discrepancy between $\delta_{\text{max load}}$ and n that is indicated is real or is due to shortcomings in the experimental technique.

The maximum load strain tends to become greatest at intermediate strain rates. This is believed to result from a lowering of $\delta_{\text{max load}}$ through creep at the minimum strain rate and adiabatic heating at the maximum strain rate.

The strain hardening characteristics of the unalloyed titanium require the use of two expressions of the form $\sigma = \sigma_0\delta^n$ and this gives rise to values of n_1 and n_2 which are plotted in Fig. 9 *versus* temperature and strain rate. It has already been pointed out that both exponents vary only slightly with test temperature and strain rate. It is evident from these data that except for the possible exception noted above, n_1 and n_2 establish the bounds for the value of $\delta_{\text{max load}}$.

The strength coefficients $\sigma_{0,1}$ and $\sigma_{0,2}$ tend to parallel the yield and tensile strengths respectively (Figs. 8 and 10). Thus with reduction in test temperature, both $\sigma_{0,1}$ and $\sigma_{0,2}$ increase, but with increase in strain rate the behavior is more complex due to the action of creep at low strain rate and adiabatic heating at high strain rates.

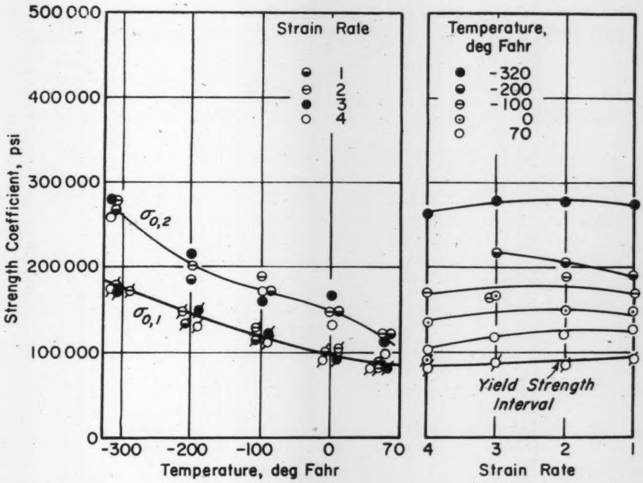


FIG. 10.—The Strength Coefficient of Unalloyed Titanium as Function of Temperature and Strain Rate. (Values averaged for lots of metal 52, 55 and AA.)

Stress-Strain Relationships in 5 Al Titanium Alloy:

The tensile and yield strengths for 5 Al titanium alloy increase uniformly with reduction in temperature (Fig. 11). As the strain rate is raised, the yield and tensile strengths increase at the maximum testing temperatures, but at −100 F and lower there is no indicated strain rate effect.

The ductility for this alloy undergoes a minimum at −200 F which probably signifies the intrusion of some phase transformation under this test condition.

The strain at maximum load is measured at values of 0.04 to 0.20 and, as is evident, this agrees with the maximum spread in n values (Fig. 12). Both n_1 and n_2 are relatively unaffected by changes in temperature and strain rate.

For the 5 Al titanium alloy the yield and tensile strengths are very nearly the same. The stress-strain curve then approximates the flow curve to be expected for an ideal plastic body. Such a material, when tested as a cylindrical specimen, has been proposed as subject to strain instability due to surface geometry effects (13). From consideration of the $\delta_{\text{max load}}$ in Fig. 12, it is evident that further complicating factors are possible temperature and strain rate changes that arise during the deformation process. For example, consider the change in the stress-strain curve as the temperature is lowered for the extreme strain rates 4 and 1.

At the minimum strain rate at 70 F two maxima arise in the stress-strain curve which consequently corresponds to

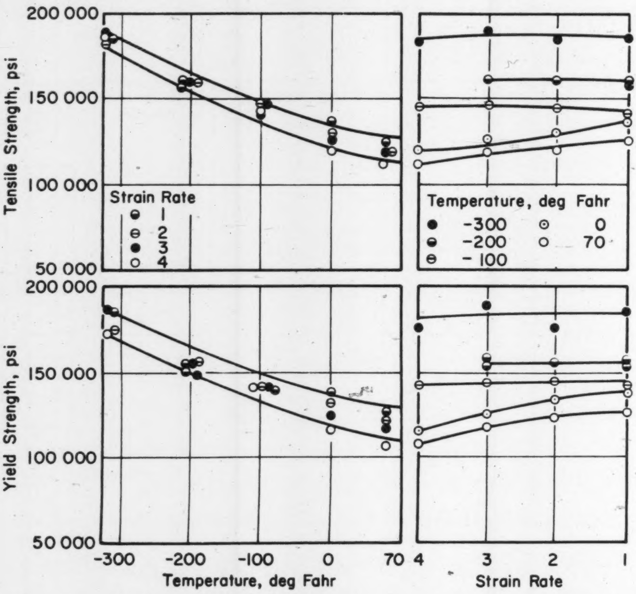


FIG. 11.—The Strength Properties of 5 Al Titanium as Function of Temperature and Strain Rate (Alloy 16).

Fig. 6(*c*). This relationship occurs at 0 F, but at −100 F the minimum in the flow curve disappears, and it then corresponds to Fig. 6(*b*). There is now but the one maximum in the curve and this is measured at all lower testing temperatures.

This behavior depends on the strain rate, and since the effect of increasing strain rate is to eliminate the minimum in the flow curve which may be considered as giving rise to the two maxima, this effect is clearly demonstrated as the strain rate changes from the minimum to the maximum rate for the alloy under examination. Thus at 70 and 0 F, as the strain rate is increased the two maxima are displaced toward each other and are indicated as becoming identical at the maximum strain rate.

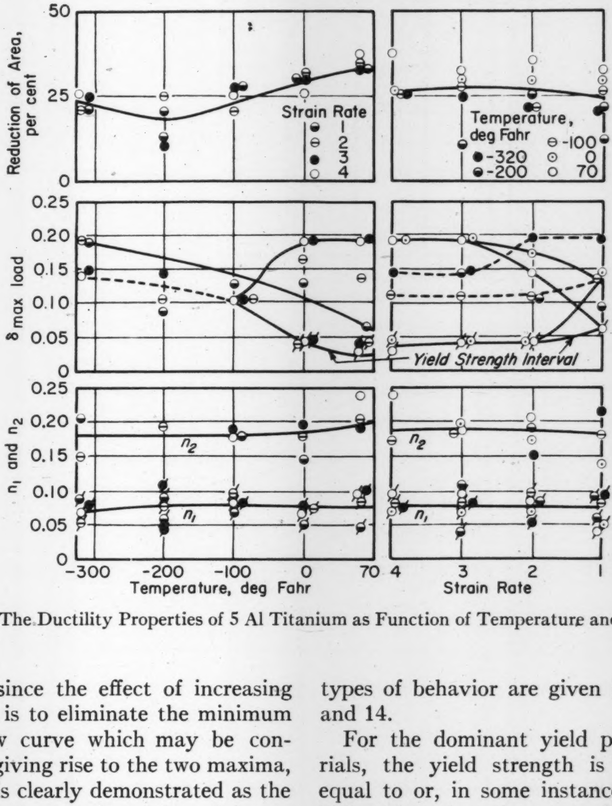


FIG. 12.—The Ductility Properties of 5 Al Titanium as Function of Temperature and Strain Rate (Alloy 16).

The Stress-Strain Relationships in 6.5 Mn Titanium Alloys:

The 6.5 Mn titanium materials were found to be separable into two groups, for the first of which a dominant yield point was observed while for the second the yield point was suppressed. Average strength properties for the respective types of behavior are given in Figs. 13 and 14.

For the dominant yield point materials, the yield strength is effectively equal to or, in some instances, slightly higher than the value entered as the tensile strength.[4] This lead to stress-strain

---

[4] For the usual definition of tensile strength this is not possible, but signifies in the present case that the first maximum was observed at a higher nominal stress than was the second maximum. The numerical difference was always small as is evident from the curves.

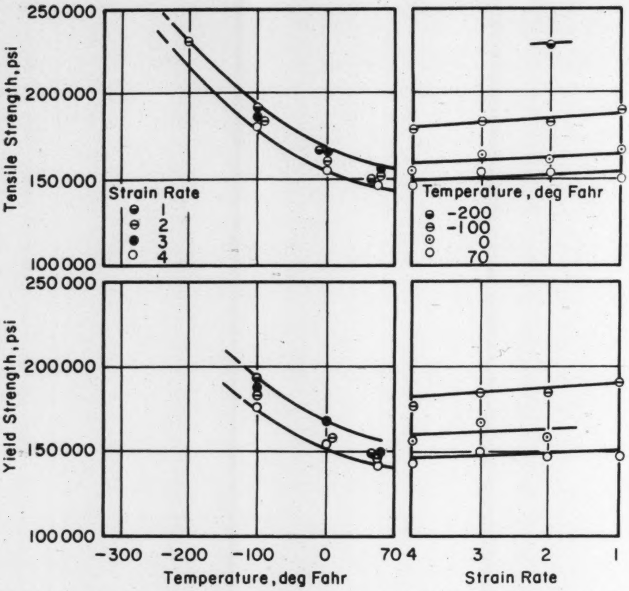


FIG. 13.—Variation of the Strength Properties for Alloy 29 (6.5 Mn Titanium) with Temperature and Strain Rate. Dominant yield point type.

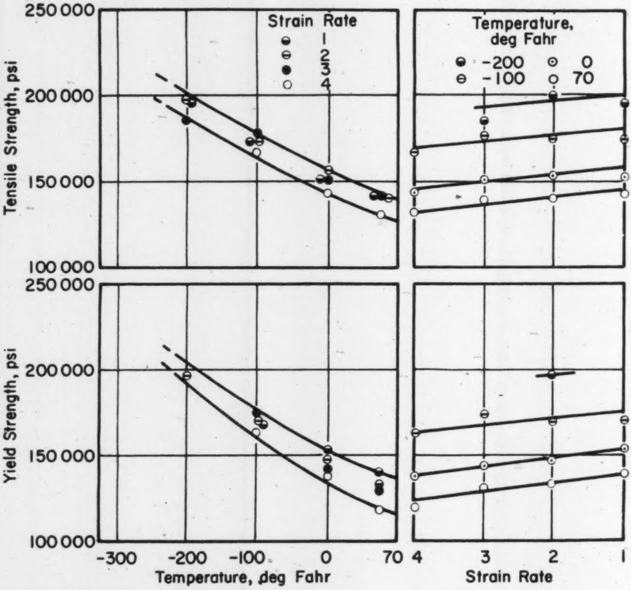
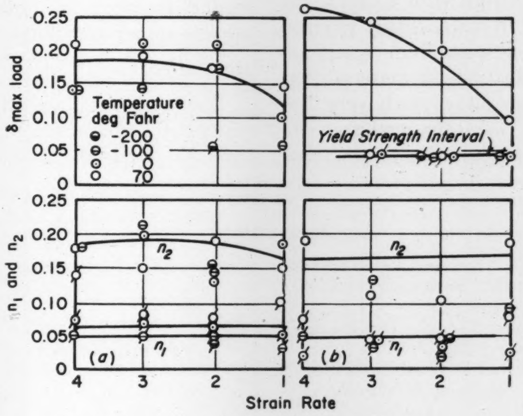


FIG. 14.—Variation of the Strength Properties for Alloy 31 (6.5 Mn Titanium) with Temperature and Strain Rate. Suppressed yield point type.

curves for which $\delta_{\text{max load}}$ effectively coincided with n_1. At the minimum strain rates a second maximum was in some instances generated, but for the most part the flow curve decreased more or less continuously to higher strains. Ductility, although low, was sufficient that a second maximum could have developed at the expected location.

The suppressed yield point type stress-strain curve was generated through an over-all lowering of that portion of the curve described by $\sigma_{0,1}\delta^{n_1}$ and not by an elevation of the high strain portion of the curve. Characteristic strength properties are presented in Fig. 14. Here, for the most part, the yield strength is less than the tensile strength.

The strains at maximum load for the two types of flow curve (Fig. 15) correspond to n_1 and n_2 respectively for the suppressed and dominant yield point curves. Differences which are attributable to the strain rate effect on $\delta_{\text{max load}}$ are also observed.



(*a*) Alloy 31; suppressed yield point type.
(*b*) Alloy 29; dominant yield point type.

FIG. 15.—Variation of $\delta_{\text{max load}}$, n_1 and n_2 as Function of Strain Rate for 6.5 Mn Titanium.

DISCUSSION AND CONCLUSIONS

The plastic deformation of titanium and titanium alloys cannot, in general, be fully described by a flow curve established by the relation $\sigma = \sigma_0\delta^n$, where the symbols have the usual significance. Rather, the stress-strain relationships are best described by a minimum of two such equations where the equations apply to low and high strain intervals, which have been designated as yield point and strain hardening intervals. The high and low strain intervals are separated, in general, by an intermediate interval. It is believed that the general behavior of titanium materials can be adequately described by suitable adjustment of these three deformation regions, where the intermediate region is constructed as the tangent maximum to the two calculated curves.

In studies of the deformation of other metals, notably magnesium, clearly established deformation ranges such as have been described for the titanium materials here investigated have been associated with changes in metallographic deformation mechanism as, for example, slip deformation at low strains and twinning at large strains. A similar explanation for the two deformation regions in titanium is possible, but a preliminary examination of the deformed tension specimens did not lead to an immediate solution to this question.

Perhaps the most important conclusion to be drawn from this work (and this serves merely to emphasize a conclusion which is implied in many earlier reports) is that high strength titanium closely approximates an ideal plastic material. Except for titanium alloys with very low yield strengths, a rather moderate increase in strain rate leads to this flow condition. For simulated ideal plastic flow the deformation interval described by $\sigma = \sigma_{0,1}\delta^{n_1}$ will govern the instability strain and this may consequently be very low. For the materials investigated the flow relations in the yield point interval are:

$$\sigma = \sigma_{0,1}\delta^{0.12} \quad \text{(unalloyed titanium)}$$

and

$$\sigma = \sigma_{0,1}\delta^{0.07} \quad \text{(alloyed titanium)}$$

For high strength titanium materials the second form of the equation probably applies rather generally.

Acknowledgment:

The authors gratefully acknowledge support for the present work under Contract No. DAI-30-115-505-ORD-686.

REFERENCES

(1) P. W. Ludwik, "Elemente der Technologischen Mechanik," Verlag Julius Springer, Berlin (1909).

(2) J. H. Hollomon, "Tensile Deformation," *Metals Technology*, Vol. 12, No. 4 (1945).

(3) J. R. Low, Jr. and F. Garafalo, "Precision Determination of Stress-Strain Curves in the Plastic Range," *Proceedings*, Soc. Experimental Stress Analysis, Vol. IV, No. II, p. 16 (1947).

(4) E. P. Klier, "The Tensile Properties of Selected Steels as a Function of Temperature," *Bulletin No. 35*, Welding Research Council, April (1957).

(5) J. H. Palm, "Stress-Strain Relations for Uniaxial Loading," *Applied Scientific Research*, Vol. A-1, p. 198 (1948).

(6) G. W. Geil and N. L. Carwile, "Tensile Properties of Copper, Nickel and Some Copper-Nickel Alloys at Low Temperature," National Bureau of Standards, *Circular 520*, p. 67 (1952).

(7) A. J. Opinsky et al, "Uniform Elongation of Titanium and Titanium Alloys," WADC TR 55-454, Part 2 (1956).

(8) G. W. Geil and N. L. Carwile, "A Reduction of Area Gage for Use at Low Temperatures," *Journal of Research*, Nationa Bureau of Standards, Vol. 43, p. 527 (1949).

(9) E. P. Klier et al, "The Properties of Constructional Metals as a Function of Temperature and Strain Rate in Torsion," WADC TR 56-216 (1956).

(10) E. P. Klier and Neil Feola, "The Effects of Interstitial Contaminants on the Notch Tensile Properties of Titanium and Titanium Alloys," WADC TR 55-325, Parts I and II (1956).

(11) G. W. Geil and N. L. Carwile, "Some Effects of Low Temperatures and Notch Depth on the Mechanical Behavior of an Annealed Commercially Pure Titanium," *Journal of Research*, National Bureau of Standards, Vol. 59, No. 3, p. 215 (1957).

(12) F. D. Rosi and F. C. Perkins, "Mechanical Properties and Strain Aging Effects in Titanium," *Transactions*, Am. Soc. Metals, Vol. 45, p. 972 (1953).

(13) V. V. Sokolovsky, "The Propagation of Elasto-Plasto Waves in Bars," *Prikladnaia Matematika i Mekhanika*, Vol. 12, p. 261 (1948)

DISCUSSION

MR. G. W. GEIL[1] (*presented in written form*).—The authors have used a tension specimen with a 4-in. radius section to localize the deformation for convenience in the placement of their diameter gage. The use of the above contoured shape has a very significant effect on the tensile behavior of unalloyed titanium specimens and the strength and ductility indices. As shown in Fig. 5 of the paper by Geil and Carwile[2] presented at ASTM Annual Meeting 1959, a radius even as large as 10 in. has an appreciable effect on the tensile behavior of unalloyed titanium; at 25 C, the notch strength was approximately 3000 psi (about 3 per cent) higher than the tensile strength of a standard unnotched specimen. Moreover, the true strain at fracture was only about 86 per cent that of the unnotched specimen. Similarly, for a specimen with a 2.0-in. radius, the notch strength was about 6000 psi (or 6 per cent) higher than the tensile strength and the true strain at fracture was only about 82 per cent that of the unnotched specimen. Interpolation from the curve for these data and those for notched specimens of smaller radii indicates that, for a 4-in. radius specimen at 25 C, the stress values would be about 5 per cent higher and the true strain at fracture values about 16 per cent lower than those of an unnotched specimen. Similar relations were obtained at 100, −78, and −196 C except that the effect of the radius on the ductility was much greater at the lower temperatures; at −196 C, the true strain at fracture of the 2.0-in. radius specimen was only about 60 per cent that of the unnotched specimen. Thus, our data on unalloyed titanium indicate that the strength and ductility values obtained by the authors must be modified appreciably if they are to be considered as representative of unnotched specimens.

True stress - true strain data obtained at the National Bureau of Standards at selected temperatures ranging from −196 to 100 C on unnotched specimens of unalloyed titanium and other metals, such as iron, copper, and nickel, provided logarithmic true stress - true strain curves that were generally sigmoidal in shape. These data could not be represented with any reasonable degree of accuracy by a combination of two straight lines.

As shown in a paper on the tensile properties of ingot iron at low temperatures,[3] the slope of logarithmic true stress - true strain curves at maximum load must be equal to the true strain at maximum load. Moreover, this relationship is entirely independent of any assumed specific true stress - true strain law—such as a power relationship of the form $\sigma = k\delta^n$, for example.

The authors state that the true strain at maximum load of the unalloyed titanium is strongly influenced by temperature, being about doubled as their temperature is reduced from 70 to −320 F. However, they show that their n_1 and n_2 values were relatively unchanged with temperature change. Thus, there apparently is no significant correlation between the strain at maximum load and their n_1 and n_2 values. This raises a question as to the value of trying to consider their nonlinear logarithmic true stress - true strain curve as being composed of two simple linear portions, representing two different power laws.

---

[1] Physicist, National Bureau of Standards, Washington, D. C.

[2] "Effect of Notch Geometry on the Tensile Properties of Annealed Titanium at 100, 25, −78, and −196 C," see p. 985, this publication.

[3] *Journal of Research*, National Bureau of Standards, Vol. 45, p. 129 (1950), (RP 2119).

MR. E. P. KLIER (*author*).—The analysis of the $\delta_{\text{max load}} = n$ relationship referred to by Mr. Geil contains an error in semantics. Thus Geil and Carwile set $\sigma = \sigma(\delta)$. Intuitively from the ln σ-ln δ plot it is then stated that

$$\begin{array}{cccc} & (1) & (2) & (3) \\ \text{slope} = & b & = \dfrac{d\,(\log_e \sigma)}{d\,(\log_e \delta)} & = \dfrac{d\sigma/\sigma}{d\delta/\delta} \end{array}$$

The generalized representation used in term (2) is dropped in term (3) and the development continues from this point in agreement with Hollomon's treatment. We maintain that the change from the generalized functional relationship in (2) to the particular relationship in (3) and subsequent development forces the σ-δ relationship to be that proposed by Ludwik. Hollomon's equation is a restricted form of Ludwik's equation. On this basis we have questioned the demonstration claimed by Geil and Carwile. That the general validity of their representation is not assured is perhaps indicated by emphasizing that $d\sigma/d\delta$ is the first derivative of $\sigma = \sigma(\delta)$ and the right side of this equation need not, in general, become equal to δ.

It is our belief that Geil and Carwile have assumed in their treatment that b in the above expression is single valued and continuous. Certainly if the yield point effects in iron are included in their generalized equation this assumption is not valid. (At each singularity in the σ-δ curve, n is discontinuous and possibly double valued.) Further this behavior is also indicated as possible at large strains, as, for example, in our Fig. 3 of the paper.

It is our belief that Geil and Carwile have stated the necessary but not the sufficient conditions for $\delta_{ML} = n$.

In Mr. Geil's last paragraph it is stated that "... there apparently is no significant correlation between the strain at maximum load and their n_1 and n_2 values." Since we have developed the contrary argument at length in the paper, we feel it necessary to assert our disagreement with this statement. But perhaps we should re-emphasize the fact that our objective in our work was to describe the stress-strain relations in the titanium studied under the conditions imposed with sufficient detail that the approximate stress-strain curve for any one given test condition could be realistically reproduced. Figures 2 to 5 were reproduced in the paper to demonstrate the agreement between the calculated and measured curves under somewhat extreme conditions. It is our belief that in the examples the agreement is good. Admittedly some error can arise when $\delta_{ML} \neq \Big|_{n_1}^{n_2}$, but falls between these extreme values. However, the error in question is very small. We maintain then that the essential details of our experimental curves can largely be reconstructed from the data presented by use of the equations and manipulations advanced. The power series representation then is meaningful and useful regardless of the agreement or not of δ_{ML} with either n_1 or n_2.

In the ln-ln plots in Figs. 2 to 5 it will be seen that, for the most part, sigmoidal shaped curves were not measured in the present work. A pronounced example of the effect as measured in the present work is illustrated in Fig. 19. In this connection we wish to point out that this effect is not new to us, and we have published curves[4] showing the same behavior reported by Geil and Carwile.

---

[4] Eugene P. Klier, "The Tensile Properties of Selected Steels as a Function of Temperature," *Bulletin*, Welding Research Council, New York, N. Y., No. 35, April 1957.

On re-examining our stress-strain curves for steels, we note that the deviation from linearity first observed by Geil and Carwile sets in at a maximum natural strain of about 0.05 and may not set in at natural strains as low as 0.01, that is, in the range 5 to 1 per cent reduction of area. For materials where the effect is most pronounced, this constitutes a relatively early portion of the plastic strain interval to maximum load, see Fig. 20. For certain of the materials studied here, the maximum figure of 5 per cent reduction of area will carry beyond the maximum load. Under this condition the sigmoidal deviation would be undetectable. The question may be considered in a different light.

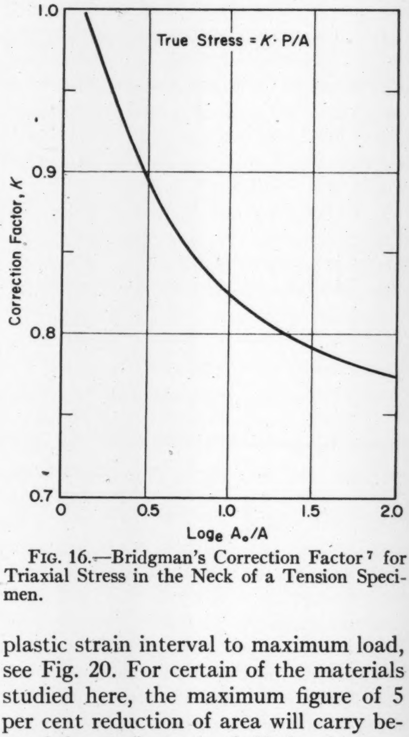


FIG. 16.—Bridgman's Correction Factor[7] for Triaxial Stress in the Neck of a Tension Specimen.

The sigmoidal deviation observed by Geil and Carwile consists effectively in the stress being measured at a low value for a specific strain. Since in their work the σ-δ curve is rising rapidly in the strain interval where the effect is measured the points calculated from $\sigma = \sigma_0\delta^n$ and those measured tend to fall in the same line but the respective points are stretched differently along the line. The deviation is scarcely evident then from inspection of the usual σ-δ plot. This deviation, it is emphasized, is to low stress values; thus the actual curve deviates to a value less than the calculated curve. In the tests which we have reported in the paper the deviations have largely been measured in the opposite direction. Stated in terms of changes in strain hardening exponent: the sigmoidal deviation tends to reduce n; in contrast the deviation which we have observed leads to an increase in n.

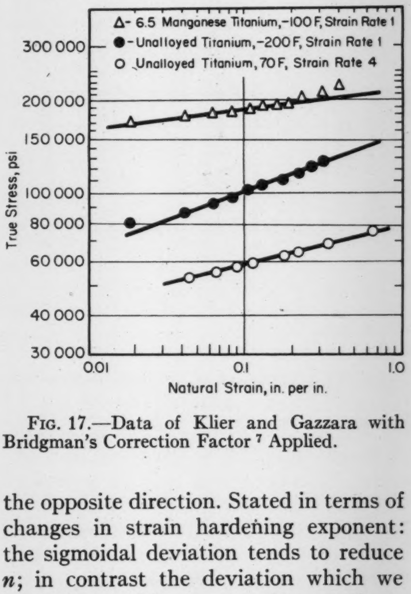


FIG. 17.—Data of Klier and Gazzara with Bridgman's Correction Factor[7] Applied.

With reference to Mr. Geil's comments re the effect of contouring the specimen on the tensile properties. We are glad to have these indicated correction factors. The contouring was necessary to allow completion of the tests under the strain rate conditions required. While it was appreciated that the contouring would affect certain of the properties measured for the specimens it is important to have the numerical magnitude of these changes which he has been able to supply from his work.

MR. C. E. FELTNER[5] (*by letter*).—The data reported by the authors is not at all surprising. The divergence of the data from the general stress - plastic strain power law has been observed for many different materials.[6] This divergence, notably in materials which exhibit ductile behavior, generally occurs slightly beyond the maximum load and is accompanied by inhomogeneous yielding or necking. For materials which exhibit relatively brittle behavior, fracture occurs before a neck develops and a single strain hardening exponent applies to the data.

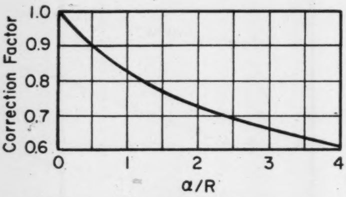


FIG. 18.—The Correction Factor K as a Function of Radius of Curvature of the Contour of the Neck (Bridgman).

In the formation of a neck, there appear radial and tangential stresses, creating a triaxial condition of stress. Several solutions have been advanced for the state of stress in the neck of a tension specimen, but the solution obtained by Bridgman[7] is probably the most accurate.

The result of this solution is a correction factor curve shown in the accompanying Fig. 16. In this figure, P is the instantaneous load, A is the instantaneous area, and A_0 is the original area. The correction factor should be applied with the cautions suggested by Bridgman.[7]

Application of this correction for inhomogeneous yielding exhibited by Figs. 3, 4, and 5 of the authors' paper, results in the curves shown in Fig. 17.

Within experimental error the data for each material and test condition may be described by a single strain hardening exponent. The room temperature data for the unalloyed titanium appears to have less scatter than the other two curves. Part of the reason for this is that the correction factor was derived without a consideration for temperature. It has also been observed that Bridgman's correction is not quite large enough. This tends to explain why the data for the top curve of Fig. 18 still diverges upward.

It is believed by the writer that the treatment given in this discussion has somewhat more tenable physical significance than does the authors' idea of using a tangent maximum to the two calculated curves constructed in the intermediate deformation region.

MESSRS. KLIER AND C. GAZZARA (*author's closure*).—Mr. Feltner's Fig. 16 taken from Bridgman's paper is derived through consideration of a precedent curve reported by Bridgman which we enter here as Fig. 18. The correction factor K which Mr. Feltner has used to replot our data is seen then to depend on the relationship α/R which was measured by Bridgman for steels in determining Fig. 16. As was pointed out by Bridgman, the validity of Fig. 16 is contingent on the validity of Fig. 18 which must be established for each new series of experiments. Mr. Feltner's discussion assumes that α/R for all specimen configurations changes so that the correction factor K decreases with increasing strain. This assumption is not valid. For a strain hardening material geometries of specimens can be so selected that through the course of the test the factor K in Fig. 16 can be

---

[5] Graduate Fellow, Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[6] J. H. Hollomon, "Tensile Deformation," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Iron and Steel Div., Vol. 162 (1945).

[7] P. W. Bridgman, "The Stress Distribution at the Neck of a Tension Specimen," *Transactions*, Am. Soc. Metals, Vol. 32, p. 553 (1944).

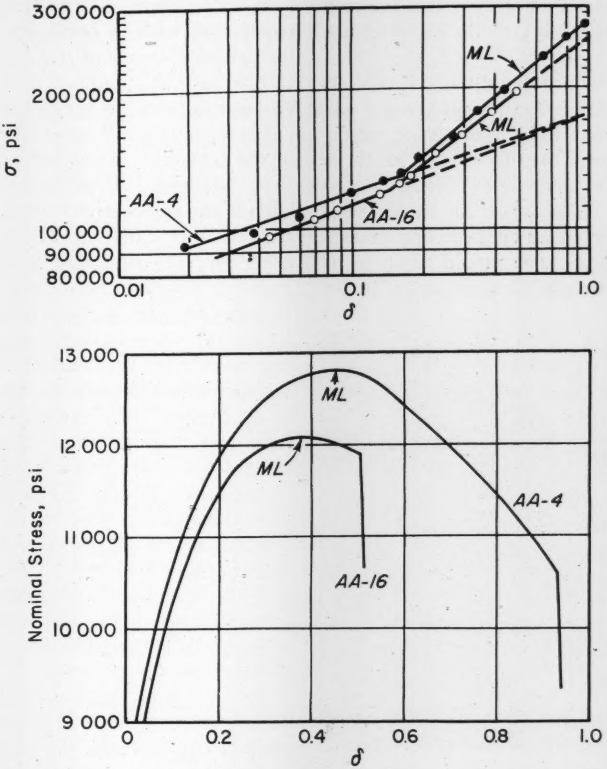


FIG. 19.—Plots of ln σ-ln δ and Nominal Stress-δ for Two Titanium Specimens. Maximum Load Points Designated ML.

TABLE II.—GEOMETRY OF TWO TITANIUM SPECIMENS TESTED AT −320 F.

Specimen	Reduction of Area, per cent	R	α	α/R Final	α/R Initial	Correction Factor, K
AA-4	72.7	0.1 in.	0.093	0.93	0.047	0.83
AA-16	41.0	~4.0 in.	0.14	0.035	0.047	0.99

α = radius of test cross-section.
R = radius of neck contour.

held nearly constant with strain increasing to the maximum load strain. Beyond this strain value the deformation process becomes unstable and K may then drop with further deformation. This is illustrated in the following tabulated data, where it is seen that for a selected test section α/R has actually decreased slightly leading to an increase in K, that is, a reduction in the stress raising potential of the section contour at maximum load. At strains beyond maximum load Bridgman's correction as advanced by Mr. Feltner potentially becomes applicable.

For the tests under discussion $\delta_{ML} \approx 0.4$, so that the σ-δ curves taken are unaffected by the Bridgman stress correction to natural strains of 0.4 in these examples. In other words according to Mr. Feltner's discussion for these specimens the discontinuity or second branch of the ln σ-ln δ curve should begin to be in evidence only at strains above 0.4. In fact this point is seen to lie between 0.15 and 0.20 for the two specimens represented in Fig. 19. This behavior then does not develop from the origin proposed by Mr. Feltner. The effect is considered by us to be real and dependent on experimental variables as we have attempted to describe in the paper. The restraint exercised by the section contour is important and should, in general, be taken into consideration. It is our belief that we have done this to a satisfactory degree and that the replot of our data as in Fig. 17 is not valid.

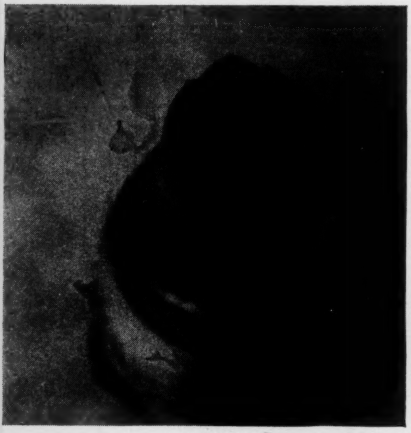


FIG. 20.—Fracture Surface of a Titanium Tension Specimen.

With reference to the Bridgman correction we wish to point out one of the disturbing features of this indicated correction is that it cannot always consistently be applied. In our earlier work with steels we found that need for application of the correction depended on the structure. It could be applied, in general, to correct the stress-strain curve for a heat-treated steel. There usually appeared no need for the correction when the test material was structural steel. In Fig. 19 for two titanium specimens the maximum load strain is indicated and it has been shown that to this point changes in α/R can produce a change in the flow stress of the order of only 1 per cent. However, from this strain to fracture strain for the specimen that undergoes necking, changes in α/R should lead to a progressive uplift in the flow curve to a maximum of nearly 20 per cent greater than that actually measured. The fact that this displacement is not observed suggests to us that physical factors not incorporated in Bridgman's analysis intrude to modify the data.

The strain to fracture phenomena in titanium are difficult to describe without lengthy discussion which is not in order here; however, we have tested standard cylindrical specimens which have deformed and remained essentially cylindrical, but of much reduced diameter at fracture. Other specimens have deformed into elliptic sections, while in one extreme material the fracture became nearly square, see Fig. 20. Outside the fracture region this specimen remained cylindrical. We plan to discuss these fracture effects and their origin at length in a future publication, but at this time we wish to reaffirm our belief that the flow properties in titanium are satisfactorily described by means of the power law relationships that we have used.

PROPERTIES OF 70-30 COPPER-NICKEL ALLOY AT TEMPERATURES RANGING UP TO 1050 F*

BY W. F. SIMMONS,[1] B. J. SIROIS,[2] D. N. WILLIAMS,[3]
AND R. I. JAFFEE[3]

SYNOPSIS

Some physical and mechanical properties for Cufenloy 30, a wrought 70.0 copper, 29.1 nickel, 0.5 iron, and 0.35 per cent manganese alloy, in the drawn and stress-relieved temper are given at elevated temperatures. The physical properties include thermal expansion, thermal conductivity, electrical resistivity and conductivity, and dynamic modulus of elasticity. The mechanical properties include the short-time tensile properties and the long-time creep and creep-rupture properties for temperatures to 1050 F.

This alloy has the proper combination of strength properties, ductility, and stress-corrosion resistance to make its use advantageous for unfired pressure vessel applications at temperatures up to 850 to 900 F.

Copper-nickel alloy tubes have been used successfully for many years in heat exchangers and unfired pressure vessels operating at elevated temperatures and pressures. Tubes in the annealed temper have invariably been used in these applications, and the design factors for the annealed tubes have been based chiefly on creep data contained in the excellent reports made in 1942 by Burghoff, Blank, and Maddigan (1),[4] Burghoff and Blank (2) in 1947, and in 1954 by Port and Blank (3).

Design factors based on annealed material have limited the 70-30 copper-nickel alloy tubes to uses and designs which have failed to take full advantage of the higher mechanical properties obtainable with this alloy. The need for improved mechanical properties in this alloy when used in heat exchangers has led to the present investigation, with the objective of producing tubes having improved properties but with sufficient ductility to permit U-bending over small radii and expansion into tube sheets.

The alloy evaluated in this investigation was Cufenloy 30 containing 70.0 copper, 29.1 nickel, 0.5 iron, 0.35 manganese, and less than 0.01 per cent lead, in the drawn and stress-relieved temper.

A considerable quantity of tubes of this alloy, process annealed to the average grain size prescribed in ASTM Specification B 111,[5] was subsequently drawn with various degrees of cold reduction to diameters ranging between $\frac{5}{8}$ and 1 in. and with wall thicknesses up to 0.120 in. Tubes in these various cold-drawn tempers were then heat treated to obtain ductility characteristics which permitted the tube to be expanded into tube sheets and U-bent over small radii.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Chief, High-Temperature Metals Research Division, Battelle Memorial Institute, Columbus, Ohio.

[2] Metallurgical Engineer, Phelps Dodge Copper Products Corp., Elizabeth, N. J.

[3] Assistant Chief and Chief respectively, Nonferrous Physical Metallurgy Division, Battelle Memorial Institute, Columbus, Ohio.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

[5] Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 58), 1958 Book of ASTM Standards, Part 2, p. 172.

It was found that tubes which conformed to the following minimum mechanical properties at room temperature were sufficiently ductile to permit the required expansion and U-bending:

Tensile strength, psi, min	72 000
Yield strength, psi, min	50 000
Elongation in 2 in., per cent, min:	
For wall thicknesses up to 0.049 in., inclusive	12
For wall thicknesses over 0.049 in.	15
Expansion of tube inside diameter with tapered pin, per cent, min	20

In addition to meeting the standard mercurous nitrate test specified in ASTM Specification B 111, specimens of tubes U-bent over a radius equal to twice the diameter of the tube have been subjected for ten months to an even more rigorous test in moist ammonia atmosphere under various conditions of stress on the outer element of the bend. No evidence of stress corrosion cracking was observed in these test specimens.

Tensile and creep properties at elevated temperatures were determined on specimens made from ¾-in. rod stock treated to develop the above minimum properties. Physical properties such as thermal expansion, thermal conductivity, and electrical resistivity were determined on specimens made from 0.900-in. rod stock of the same material.

PROCEDURES

Thermal Expansion:

Linear-thermal-expansion measurements were made in a vertical-quartz-tube dilatometer in which dilations were measured by a calibrated dial indicator that has divisions of 0.0001 in. The measurements were made with the specimens in helium at atmospheric pressure. A 3-in.-long cylindrical specimen was used.

Length changes were read from the dial indicator at the same time that temperatures were measured. Each specimen was given two thermal cycles from room temperature to 750 F and back to room temperature. A best curve was drawn through the experimental points, and mean-linear-thermal-expansion coefficients were interpolated over selected temperature ranges from this curve.

The error of the results is estimated not to exceed plus and minus 2 per cent.

Thermal Conductivity:

The apparatus and method used in making the thermal-conductivity measurements are essentially the same as in the steady-heat-flow method described by M. S. Van Dusen and S. M. Shelton (4). The method consists in heating one end of a specimen, measuring the temperature gradients along the specimen, and determining the rate of heat flow through the specimen by means of an iron standard of known thermal conductivity attached to the cold end of the specimen. Radial heat flow into, or away from, the specimen is minimized by thermal insulation and an encircling guard tube in which temperatures are adjusted, as nearly as possible, to match those in the specimen and standard at corresponding levels.

Five 36-gage chromel alumel thermocouples were wedged in holes equally spaced along the specimen, and two similar thermocouples were placed in the ingot iron standard. This permitted the calculation of four thermal-conductivity values, each at a different mean temperature, for each thermal equilibrium. Four thermal equilibria were obtained over a temperature range of approximately 90 to 575 F. A best curve was drawn through these four points, and the thermal conductivity was interpolated at selected temperatures from this curve.

The maximum error of the thermal

conductivity values is estimated not to exceed ±5 per cent, the chief uncertainty being the thermal conductivity of the ingot iron standard.

Electrical Resistivity:

Electrical resistivity measurements were made by the voltage-drop method with the specimens protected by a vacuum of approximately 5×10^{-3} mm of mercury. A direct current was passed through the specimen, and voltage drop was measured over a section of the specimen approximately 2 in. long. Measurements were taken with the current flowing normally and reversed, and the values were averaged to minimize thermal and induced potentials.

The error of the electrical resistivity values is estimated not to exceed ±1 per cent.

Dynamic Modulus of Elasticity:

Dynamic modulus of elasticity measurements were made by suspending a slender cylindrical specimen of the test material horizontally in a tubular furnace and causing it to vibrate transversely (in the vertical plane). Its fundamental natural frequency was then determined through location of the proper resonance peak of vibration. From this frequency, and the mass and dimensions of the test bar, the modulus of elasticity was calculated. The procedure used was based on the work of Roberts and Nortcliffe (5) in 1947.

Tensile Properties:

Tensile properties were determined between 75 and 1050 F by using standard 0.505-in. in diameter threaded-end tension specimens. Duplicate or triplicate tests were run at each temperature examined. The specimens were heated to 300 F by immersing them in heated oil during testing. Above 300 F, an electrical-resistance furnace was used to heat the specimens. The atmosphere was static air. The specimen temperature was measured by means of thermocouples attached to the specimen.

Stress-strain curves were obtained by means of a Baldwin high-magnification extensometer, microformer type, and a standard Baldwin-Southwark Tate-Emery testing machine with recorder. Strain gages (SR-4 type) were used at 75 F.

All specimens were tested at strain rates of 0.001 in. per in. per min up to the yield strength and 0.05 in. per in. per min from the yield strength to fracture.

Creep and Stress-Rupture Properties:

Creep and stress-rupture properties were determined between 500 and 1050 F by using the same type of specimen as used in the tensile property studies. Loading was accomplished in lever-arm creep machines (9:1 lever-arm ratio), with the specimens heated in an electrical-resistance furnace. The atmosphere was static air. The desired test temperature was maintained within ±2 F. Tests were conducted in accordance with ASTM Recommended Practice E 22.[6]

Creep was measured optically through windows in the furnace by means of two platinum-strip extensometers on opposite sides of the specimens and a filar microscope. Each reading was made by two observers, and the results were averaged. With this arrangement, creep movements as small as 0.0022 per cent were measured.

Specimens were held 1 hr at temperature before loading. The zero reading for creep was obtained at temperature before application of the load. Both creep strain and time to rupture, if failure oc-

---

[6] Recommended Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 – 41), 1955 Book of ASTM Standards, Part 1, p. 1605. (*Discontinued—replaced by Rec. Practice E 139.*)

TABLE I.—MEAN-LINEAR-THERMAL-EXPANSION COEFFICIENTS OF CUFENLOY 30.

Temperature Range, deg Fahr	Mean Linear Thermal Expansion Coefficients per deg Fahr over the Temperature Range Shown
68 to 200	8.7×10^{-6}
68 to 300	8.9
68 to 400	9.1
68 to 500	9.2
68 to 600	9.3
68 to 700	9.4
68 to 750	9.4

TABLE II.—THERMAL CONDUCTIVITY OF CUFENLOY 30.

Temperature, deg Fahr	Thermal Conductivity, Btu per sq ft per hr per deg Fahr per ft
68[a]	16.8
100	17.0
200	17.8
300	18.8
400	19.8
500	21.0
572	22.0

[a] Extrapolated.

TABLE III.—ELECTRICAL RESISTIVITY AND ELECTRICAL CONDUCTIVITY OF CUFENLOY 30.

Temperature, deg Fahr	Electrical Resistivity, ρ, μohm-cm	$\frac{\rho t}{\rho 68}$[a]	Electrical Conductivity, σ, megmho-cm[b]
68	36.3	1.00	0.0276
100	36.5	1.01	0.0274
200	36.8	1.01	0.0272
300	37.0	1.02	0.0270
400	37.3	1.03	0.0268
500	37.5	1.03	0.0267
572	37.6	1.04	0.0266

[a] $\frac{\rho t}{\rho 68} = \frac{\text{Electrical resistivity of temperature}}{\text{Electrical resistivity at 68 F}}$.

[b] megmho-cm = $(\mu\text{ohm-cm})^{-1}$.

curred at the stress level examined, were measured. The creep and rupture testing equipment and the procedure used have been described more fully in a previous paper (6).

TEST RESULTS

Physical Properties:

The mean-linear-thermal-expansion coefficients of Cufenloy 30 at selected temperature intervals between 68 and 750 F are given in Table I. Thermal conductivity values are given in Table II. The electrical resistivity data are reported in Table III. These data have also been given in terms of electrical conductivity. At 68 F, the electrical conductivity of Cufenloy 30 is 4.8 per cent of that of the International Annealed Copper Standard. The dynamic modulus of elasticity data are given in Table IV.

TABLE IV.—DYNAMIC MODULUS OF ELASTICITY OF CUFENLOY 30.

Temperature, deg Fahr	Dynamic Modulus of Elasticity, psi
80	20.9×10^{6}
150	21.0
200	20.9
420	20.2
615	19.4
800	18.8
1000	17.7
80	21.1

The dynamic modulus specimen was 6.25 in. long, 0.250 in. in diameter, and weighed 44.85 g.

Tensile Properties:

The short-time tensile data obtained for Cufenloy 30 are given in Table V. The yield strength was measured both at 0.2 per cent offset and 0.5 per cent extension under load. An examination of Table V will reveal that these two criteria yield very similar values. Proportional limit, tensile strength, elongation, and reduction of area were also determined.

The tensile strength and the 0.5 per cent extension yield strength have been plotted as a function of temperature in Fig. 1.

TABLE V.—TENSILE PROPERTIES OF CUFENLOY 30.

Temperature, deg Fahr	Proportional Limit, psi	Yield Strength, psi		Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
		0.2 per cent Offset	0.5 per cent Extension			
75	49 100	62 100	62 100	76 800	26	67
75	41 800	60 600	61 400	76 100	25	68
102	45 500	62 000	62 000	76 100	24	64
99	47 200	62 100	62 400	76 600	24	66
100	39 700	59 500	59 700	74 600	26	66
152	42 000	61 000	61 300	71 900	24	64
151	46 400	60 600	59 900	74 600	24	66
151	44 600	59 200	59 500	73 600	25	69
199	44 900	59 900	60 400	73 100	24	66
201	43 300	59 400	59 400	72 100	24	67
202	41 800	60 100	60 400	73 100	23	67
249	43 800	57 800	58 000	71 600	22	65
249	43 400	57 400	57 700	70 600	24	67
251	41 700	59 200	59 700	71 900	22	68
300	40 300	57 000	57 000	69 600	24	68
299	41 800	55 200	55 500	68 600	23	67
299	43 600	58 300	58 500	69 900	22	68
360	38 300	54 900	55 200	67 000	22	64
342	45 600	58 400	58 500	68 600	22	64
350	44 700	56 300	56 500	67 600	22	68
398	43 700	57 000	57 000	67 100	21	63
408	38 800	56 900	57 200	66 400	21	66
400	41 600	55 700	55 900	67 300	22	65
447	36 400	49 800	50 200	65 400	20	66
460	41 900	54 400	54 600	64 700	21	66
443	43 400	55 000	55 500	65 700	22	67
507	35 200	46 700	47 200	63 200	21	64
500	44 100	56 900	56 600	65 000	22	67
495	40 300	53 300	53 600	63 700	21	65
557	43 200	53 700	53 700	61 000	20	66
535	42 400	56 500	56 700	61 900	19	63
537	40 800	54 300	54 600	64 400	20	64
603	32 800	52 000	51 800	62 000	18	63
610	40 300	52 200	52 500	61 000	20	65
617	43 200	56 000	56 100	62 100	19	61
665	37 200	52 500	52 900	62 000	20	63
647	39 600	53 300	53 900	61 900	20	63
642	39 600	52 700	53 100	62 000	18	65
699	46 600	52 600	53 500	61 500	19	62
707	40 400	53 100	53 500	61 100	19	62
705	40 200	53 700	54 200	62 000	19	61
745	35 600	53 000	53 000	60 000	18	60
752	43 400	53 200	53 600	61 500	19	63
747	38 200	51 100	51 300	60 400	18	60

[Continued on page 1040.]

TABLE V.—*Concluded.*

Temperature, deg Fahr	Proportional Limit, psi	Yield Strength, psi		Tensile Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
		0.2 per cent Offset	0.5 per cent Extension			
847	37 300	50 000	51 100	57 400	22	64
852	31 400	50 200	50 500	56 700	22	62
952	29 800	47 500	47 400	50 700	21	49
942	38 300	47 800	47 600	51 500	21	50
1042	21 200	40 700	40 700	42 900	16	48
1047	20 600	40 300	40 500	42 100	15	45

NOTE.—The modulus of elasticity at 75 F was 22,000,000 psi.

Creep and Stress-Rupture Properties:

Results of creep studies of Cufenloy 30 are given in Table VI. Both minimum creep rate and total strain as a function of time are included. Stress-rupture properties of Cufenloy 30 are given in Table VII. Ductility at fracture is reported as is time to rupture. The minimum creep rate was also determined during the stress-rupture tests and is given in Table VII.

The data contained in Tables VI and VII have been used to construct the graph shown in Fig. 2, which describes the creep and rupture characteristics of Cufenloy 30. The stress-creep rate and stress-rupture time curves were not straight lines on logarithmic coordinates. This behavior is not unusual for copper alloys.

It was observed that at stresses where both creep and rupture data were available, the stress-creep rate and the stress-rupture time curves were very nearly

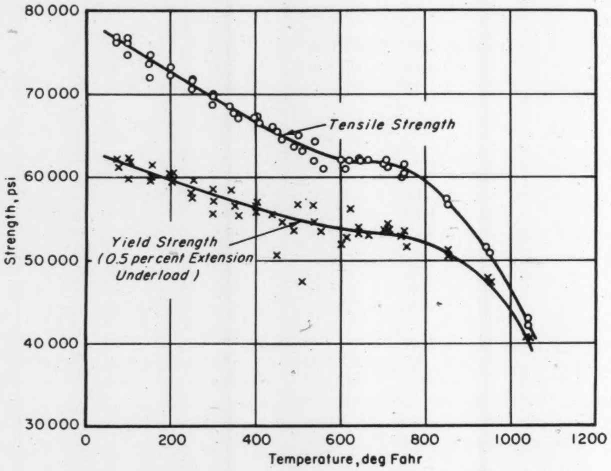


FIG. 1.—Tensile and Yield Strengths of Cufenloy 30.

TABLE VI.—CREEP PROPERTIES OF CUFENLOY 30.

Temperature, deg Fahr	Stress, psi	Duration, hr	Minimum Creep Rate, per cent per hr	Initial Strain, per cent	Total Strain in Indicated Time, per cent				
					500 hr	1000 hr	1500 hr	2000 hr	2500 hr
500	60 000	342.7	0.000017[a]	2.209	...	...	...	...	...
	50 000	282.1	0.00003[a]	0.342	...	...	...	...	...
	40 000	214.9	Nil	0.225	...	...	...	...	...
600	55 000	532.7	0.00008[a]	0.803	0.940	...	...	...	...
	50 000	817.2	0.00002[a]	0.356	0.426	...	...	...	...
	40 000	478.4	0.000015[a]	0.226	0.255[b]	...	...	...	...
	30 000	195.7	Nil	0.174	...	...	...	...	...
750	40 000	1391.3	0.00040	0.274	0.620	0.818	...	...	...
	35 000	933.4	0.00017	0.214	0.391	0.490[b]	...	...	...
	30 000	1367.9	0.000055	0.175	0.302	0.332	0.359[b]	...	...
	25 000[c]	2351.8	0.000032	0.153	0.241	0.262	0.283	0.303	0.319[b]
	18 000	2516.5	0.000015	0.118	0.176	0.192	0.204	0.212	0.219
850	25 000	1014.2	0.00061	0.167	0.687	0.993	...	...	...
	14 000[c]	1724.6	0.000072	0.090	0.258	0.304	0.339	...	...
	7 000	2512.2	0.000019	0.034	0.090	0.108	0.123	0.133	0.142
950	6 000	1104.3	0.00018	0.042	0.204	0.292	...	...	...
	2 000	1491.2	0.000032	0.018	0.061	0.075	0.096	...	...
1050	1 500	746.9	0.0003	0.015	0.185	...	...	...	...

[a] Lowest creep rate within duration of test.

[b] Extrapolated value.

[c] A room-temperature tension test was made on this specimen after creep testing.

TABLE VII.—STRESS-RUPTURE PROPERTIES OF CUFENLOY 30.

Temperature, deg Fahr	Stress, psi	Rupture Time, hr	Minimum Creep Rate, per cent per hr	Initial Strain, per cent	Total Strain in Indicated Time, per cent			Elongation in 2 in., per cent	Reduction of Area, per cent
					500 hr	1000 hr	1500 hr		
750	55 000	22.9	0.20	1.79	...	...	...	21.0	63.3
	50 000	467.9	0.0074	0.380	...	...	...	20.1	49.6
	45 000	2124.4	0.0010	0.330	1.05	1.83	3.25	17.5	31.0
850	45 000	29.2	0.13	0.391	...	...	...	25.1	44.8
	40 000	96.7	0.029	0.302	...	...	...	18.9	39.6
	35 000	469.4	0.007	0.254	...	...	...	18.4	29.2
	30 000	1539.7	0.0022	0.230	1.59	3.60	9.80	12.9	21.2
950	35 000	11.8	0.35	0.353	...	...	...	16.6	34.5
	27 000	117.9	0.044	0.195	...	...	...	15.9	35.4
	18 000	1839.6	0.0034	0.150	2.05	4.95	11.2	33.7	32.9
1050	24 000	25.5	0.28	0.246	...	...	...	34.8	41.5
	19 000	82.6	0.065	0.176	...	...	...	18.6	42.0
	10 000	968.6	0.007	0.110	7.20	...	...	59.7	53.0

mirror images of each other. This observation was applied in extrapolating the stress-rupture time curves from the longest rupture times, about 2000 hr, to determine estimated stress for rupture in 100,000 hr. The procedure used for extrapolation is in accordance with the work of Monkman and Grant (7) who showed that

$$\log t_r + m \log (mcr) = C \ldots\ldots (1)$$

where:

t_r = the rupture time,
mcr = the minimum creep rate, and
m and C = constants.

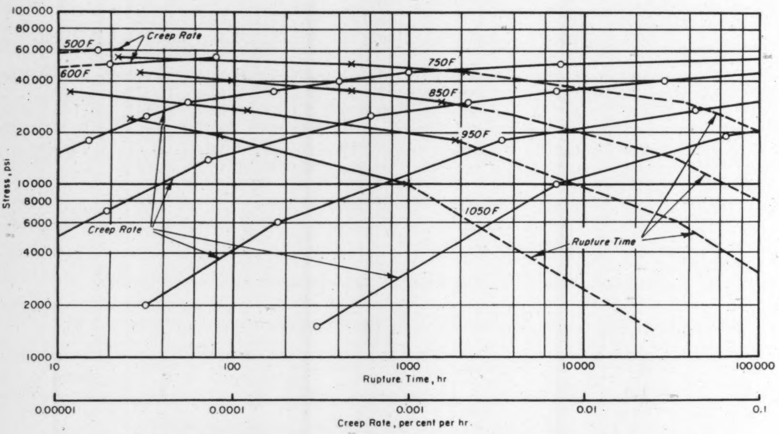


FIG. 2.—Creep and Rupture Characteristics from 500 to 1050 F for Cufenloy 30.

A plot of rupture time *versus* minimum creep rates for Cufenloy 30 showed that the slope m in Eq 1 is approximately unity, and therefore the product of the creep rate and the rupture time is constant. Since this relationship is approximately true for Cufenloy 30, the rupture time curves in Fig. 2 have been extrapolated using the negative slope of the creep rate curves. This method of extrapolation is much more conservative than a straight-line extrapolation of a log-log plot of the rupture data. It is also more conservative than an extrapolation by means of the Larson-Miller (8) parameter:

$$P = T(C + \log t) \ldots\ldots\ldots (2)$$

where:

T = absolute temperature, deg Rankin,
t = rupture time, hr, and
C = constant.

when either values of C = 20 or C = 15 are used.

DISCUSSION OF RESULTS

The ASME Boiler and Pressure Vessel Code[7] has formulated a "Basis for Establishing Stress Values for Nonferrous Materials" which is used in conjunction with past experience and knowledge of similar materials as the criteria for selecting maximum allowable stress values. An excerpt from these criteria, as outlined in Appendix Q of Section VIII of "Rules for Construction of Unfired Pressure Vessels" (1956), is as follows:

(*a*) Except for bolting materials, the maximum allowable stress values may be determined as the lowest of the following when the tensile and yield strengths are obtained from standard short-time tests:

---

[7] Report of Subcommittee of Boiler and Pressure Vessel Committee, Am. Soc. Mechanical Engrs., New York, N. Y. (1956).

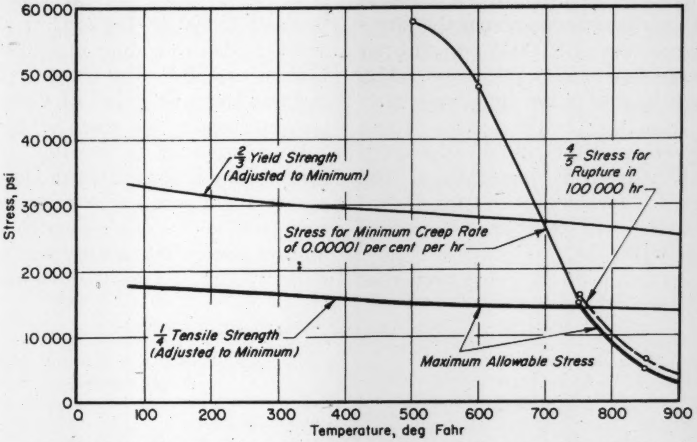


FIG. 3.—Basis for Establishing Maximum Allowable Stress for Cufenloy 30.

(1) one-fourth of the tensile strength, as adjusted to minimum;
(2) two-thirds of the yield strength (as defined in the material specification) as adjusted to minimum;
(3) the stress producing a secondary creep rate of 0.1 CRU (creep rupture units) or 0.1 per cent in 10,000 hr (0.00001 per cent per hr); and
(4) four-fifths of the stress producing rupture in 100,000 hr.

(*c*) The tests are made at the temperature under consideration.

(*d*) The tensile and yield strength are adjusted to minimum by multiplying the test results by the ratio:

$$\frac{\text{(minimum specified or expected tensile or yield strength)}}{\text{(actual room-temperature tensile or yield strength for the lot of material under test)}}$$

TABLE VIII.—MAXIMUM ALLOWABLE STRESS FOR CUFENLOY 30.*

Temperature, deg Fahr	Tensile Strength, psi (adjusted to minimum)[a]	Stress for Creep Rate of 0.1 Per Cent in 10,000 hr	Maximum Allowable Stress[b], psi	Copper-Nickel 70-30 Annealed Maximum Allowable Stress,[c] psi
75.....	72 000	...	18 000	...
150.....	69 900	...	17 500	11 600
300.....	65 400	...	16 400	10 600
450.....	61 500	...	15 400	10 100
600.....	58 400	48 000	14 600	9 600
700.....	57 800	26 500	14 400	9 400
750.....	57 400	15 000	14 400	...
850.....	53 900	5 000	5 000	...
950.....	...	800	800	...

* Determined in accordance with Appendix Q, Section VIII, of ASME Boiler and Pressure Vessel Code.

[a] Data from Fig. 1 multiplied by 0.942.

[b] One-fourth of tensile strength (adjusted to minimum) or stress to give creep rate of 0.1 per cent strain in 10,000 hr, whichever is lower.

[c] From Table UNF 23, ASME Boiler and Pressure Vessel Code. Section VIII.

The data presented in this paper have been used to determine the maximum allowable stress for Cufenloy 30 based on the above criteria, and these data are plotted in Fig. 3. To adjust the yield and tensile strengths to minimum, the tensile strengths in Table IV were multiplied by 0.942 and the yield strengths by 0.809. These adjustment factors were calculated using the minimum room-temperature properties given at the beginning of this paper. It is interesting to note that even though the stress for rupture in 100,000 hr was extrapolated by a very conservative method, four-fifths of the rupture strength yields stresses above the stress for a creep rate of 0.00001 per cent per hr. The maximum allowable stress in Fig. 3, shown by the heavy line, was determined from the tensile strength up to slightly above 750 F and by the creep strength above this temperature. The maximum allowable stresses for Cufenloy 30 from 75 to 950 F are summarized in Table VIII.

A comparison of the creep properties of Cufenloy 30 and annealed 70-30 copper-nickel alloy as reported by Port and Blank (3) and by Jenkins and Johnson (9) show that the strength advantages of the drawn and stress-relieved temper will be maintained up to temperatures in the range of 850 to 900 F. This comparison is based on the creep strength at a creep rate of 0.0001 per cent per hr, because data at 0.00001 per cent per hr were not available for comparison purposes. Table VIII also shows the boiler code maximum allowable stresses for annealed copper-nickel 70-30 alloy.

Room-Temperature Tensile Properties of Specimens Subjected to Creep Testing:

To determine the effect that long-time exposure to stress and temperature has on the room-temperature tensile properties, two creep-tested specimens were subsequently tested in short-time tension. One specimen had been exposed to a stress of 25,000 psi for 2352 hr at 750 F and the other to a stress of 14,000 psi for 1725 hr at 850 F. The tensile properties of these specimens, and of Cufenloy 30 before exposure, are given in Table IX.

Prior exposure to elevated temperatures during creep testing produced only a very slight increase in room temperature yield and tensile strengths. No significant change in ductility was noted in either specimen. Thus thermal instability is not expected to be a problem in this alloy.

TABLE IX.—ROOM-TEMPERATURE TENSILE PROPERTIES AFTER CREEP EXPOSURE.

Creep Exposure				Tensile Properties				
				Yield Strength, psi		Ultimate Tensile Strength, psi	Elongation, per cent	Reduction of Area, per cent
Time, hr	Temperature, deg Fahr	Stress, psi	Strain, per cent	0.2 per cent Offset	0.5 per cent Extension			
None	...	...	...	62 100	62 100	76 800	26	67
None	...	...	...	60 600	61 400	76 100	25	68
2352	750	25 000	0.3	64 600	64 700	79 100	26.5	68
1725	850	14 000	0.3	63 100	63 900	77 500	25	64

CONCLUSIONS

The data presented in this paper indicate that Cufenloy 30 has the proper combination of strength properties, ductility, and stress-corrosion resistance to make its use advantageous for heat exchanger applications. The drawn and stress-relieved temper of this alloy produces superior strength properties which are maintained up to temperatures in the range 850 to 900 F. This alloy meets the need for improved mechanical properties in the 70-30 copper-nickel alloy composition, and it is believed that the drawn and stress-relieved temper should be considered for inclusion in a revision of ASTM Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111).

Acknowledgment:

The authors wish to acknowledge the interest and support of the investigation in which these results were obtained by Mr. J. J. Conlon, Vice-President, Phelps Dodge Copper Products Corp., and the assistance of Mr. W. T. Erickson who was responsible for the mill development work on the alloy, and Mr. L. Caruso under whose direction the stress-corrosion tests were made. On the Battelle staff, Mr. H. W. Deem and Mr. E. H. Eldridge made the physical measurements, Mr. R. A. Wood the tension tests, and Mr. D. P. Moon the creep and stress-rupture tests.

REFERENCES

(1) H. L. Burghoff, A. I. Blank, and S. E. Maddigan, "The Creep Characteristics of Some Copper Alloys at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 668 (1942).

(2) H. L. Burghoff and A. I. Blank, "The Creep Characteristics of Copper and Some Copper Alloys at 300, 400, and 500 F," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 725 (1947).

(3) J. H. Port and A. I. Blank, "The Creep Characteristics of Copper-Nickel Alloys at 300, 400, and 500 F," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 1038 (1954).

(4) M. S. Van Dusen and S. M. Shelton, "Apparatus for Measuring Thermal Conductivity of Metals Up to 600 C," *Journal of Research*, National Bureau of Standards, Vol. 12, pp. 429–440 (1934).

(5) M. Roberts and J. Nortcliffe, "Measurement of Young's Modulus at High Temperatures," *Journal*, Iron and Steel Inst., Vol. 157, Nov., 1947, p. 345.

(6) W. F. Simmons and J. G. Kura, "The Creep Properties of Three Low-Shrinkage, Copper-Base Casting Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 791 (1958).

(7) F. C. Monkman and N. J. Grant, "An Empirical Relationship Between Rupture Life and Minimum Creep Rate in Creep-Rupture Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 593 (1956).

(8) F. R. Larson and J. Miller, "A Time-Temperature Relationship for Rupture and Creep Stresses," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 74, p. 765 (1952).

(9) W. D. Jenkins and C. R. Johnson, "Creep of Annealed Nickel, Copper, and Two Nickel-Copper Alloys," *Journal of Research*, National Bureau of Standards, Vol. 60, March, 1958, p. 3.

(10) Cyril Stanley Smith and Earl W. Palmer, "Thermal and Electrical Conductivities of Copper Alloys," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. of Metals Div., Vol. 117, p. 225 (1935) (TP-648).

DISCUSSION

Messrs. H. L. Burghoff, P. Kuzmenko, and V. F. Nole[1] (*presented in written form*).—One of the most stable copper-base alloys is 70-30 copper-nickel. Condenser and heat-exchanger tubes of this alloy have long been used in high-quality units. In such installations the tube has almost always been in the annealed condition, meeting the requirements of ASTM Specification B 111.[2] Quite recently, however, a desire has been expressed for 70-30 copper-nickel tube in a harder and stronger temper which will permit higher service stress, particularly in feedwater heaters. The present work on rod provides a good basis for selection of allowable design stresses for the drawn-and-stress-relieved condition. Work at Chase Brass and Copper Co. on tube in such a temper further emphasizes the practicability of so doing. Some of the information obtained from this work is presented herewith.

The primary reason for applying a stress-relief anneal to tubes of the alloy is to obtain greater ductility than is present in the tube as drawn and at the same time to retain essentially the greater strength of the drawn condition. The making of U-bends and even the roller expansion into tube sheets is thereby facilitated. Figures 4 to 6 show the room-temperature tensile properties of $\frac{5}{8}$-in. outside diameter by 0.069-in. wall tube as drawn, with 30, 40, and 49 per cent reductions of area and then heated for periods of 1 hr at temperatures up to 1200 F. All of this material originated from one extrusion billet of the following composition: 68.42 copper, 30.11 nickel, 0.58 iron, 0.54 manganese, 0.013 lead, 0.42 zinc.

Tensile strength as a function of annealing temperatures is given in Fig. 4. It is seen to increase at first and then to decrease as annealing temperatures increase above 600 F. Metallographic examination of all three tempers showed no structural change from the drawn condition after the 1-hr anneals up to and including 1100 F. As annealed at 1150 F, the tube drawn 30 per cent still showed no evidence of recrystallization, but that drawn 40 per cent was about 80 per cent recrystallized. As annealed at 1200 F, the tube drawn 30 per cent was about 50 per cent recrystallized and those drawn 40 per cent and 49 per cent were completely recrystallized.

The correlation of yield strength with annealing temperature is given in Fig. 5 and is similar to that for tensile strength.

Figure 6 provides the corresponding data on elongation, which is a property of great significance in the making of U-bends. The tube as drawn 30 per cent has distinctly greater ductility than those drawn 40 per cent and 49 per cent and maintains this relationship throughout the stress-relieving range. Indeed, for anneals of 800 F to 1150 F, the elongation for this temper is sufficiently high to make U-bends of small radius, while

---

[1] Assistant Director Research and Development, Physical Metallurgist, and Corrosion Engineer, respectively, Chase Brass and Copper Co., Inc., Waterbury, Conn.

[2] Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 58), 1958 Book of ASTM Standards, Part 2, p. 172.

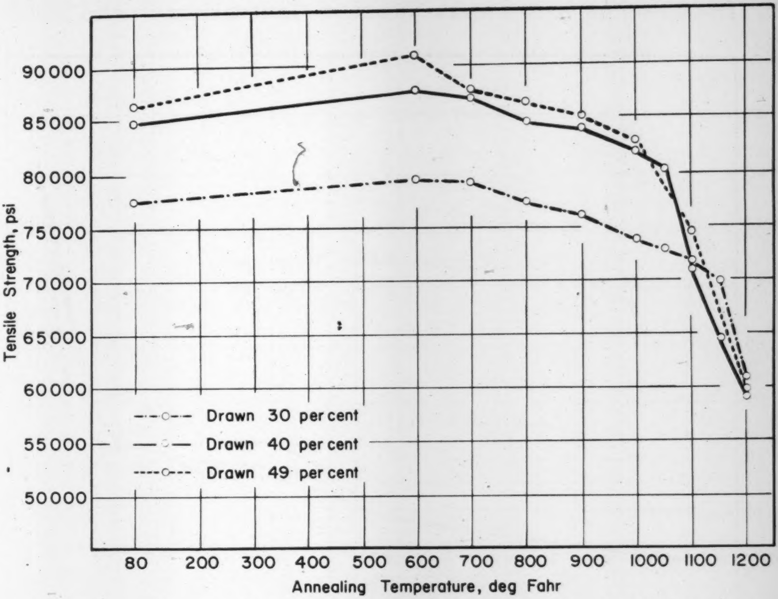


FIG. 4.—Tensile Strength and Annealing Temperature Relationships for 70-30 Copper-Nickel Tubes ($\frac{5}{8}$-in. outside diameter by 0.069-in. wall) in Three Drawn Tempers.

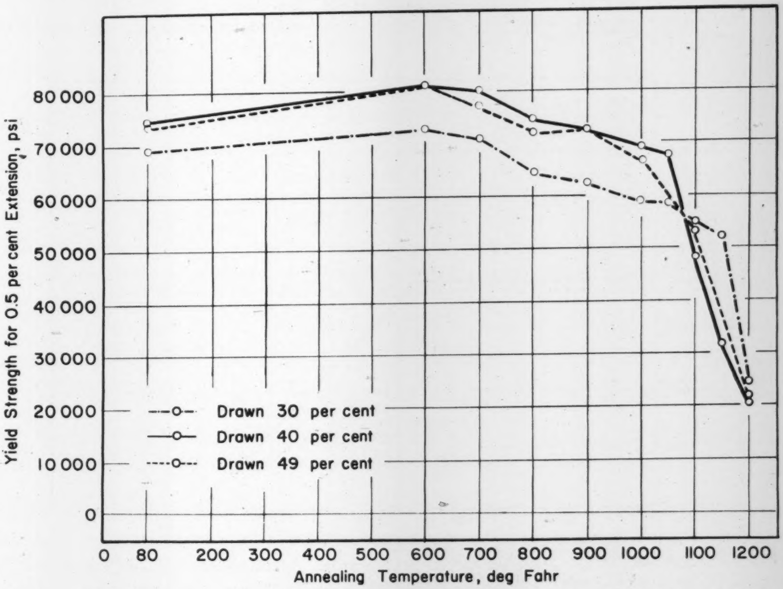


FIG. 5.—Yield Strength and Annealing Temperature Relationships for 70-30 Copper-Nickel Tubes ($\frac{5}{8}$-in. outside diameter by 0.069 in. wall) in Three Drawn Tempers.

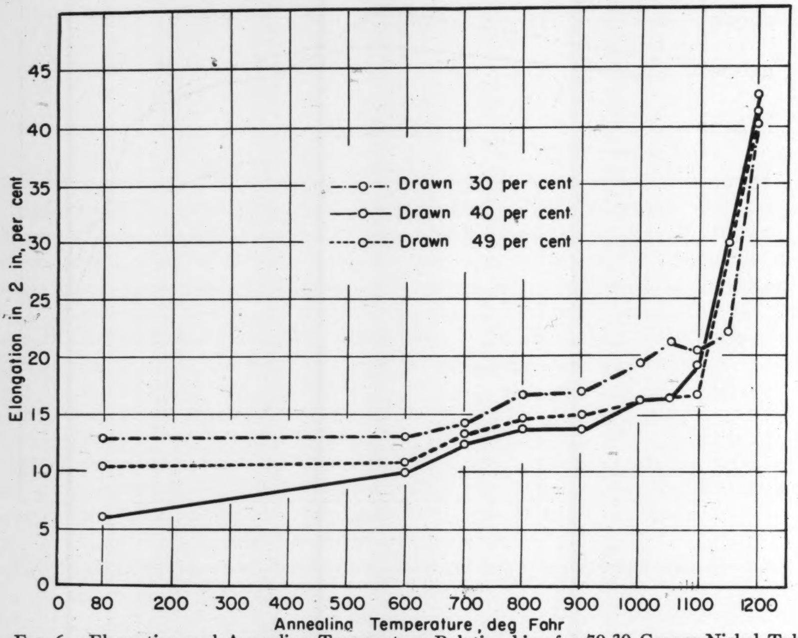


FIG. 6.—Elongation and Annealing Temperature Relationships for 70-30 Copper-Nickel Tubes ($\frac{5}{8}$-in. outside diameter by 0.069-in. wall) in Three Drawn Tempers.

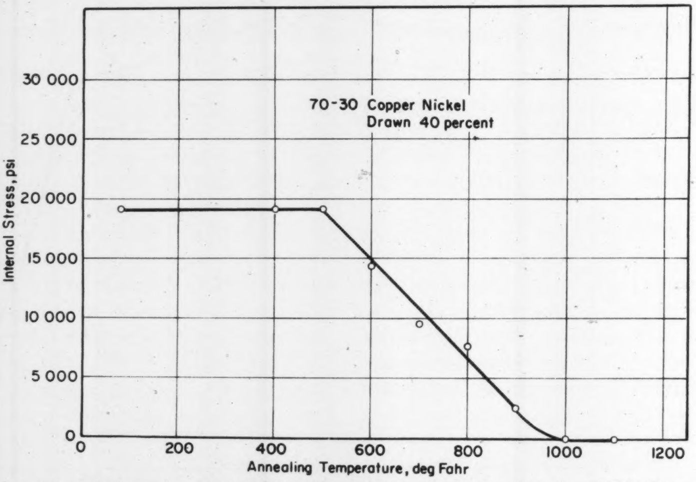


FIG. 7.—Internal Stress* and Annealing Temperature Relationships for 70-30 Copper-Nickel Tubes ($\frac{5}{8}$-in. outside diameter by 0.069-in. wall) Drawn 40 per cent.

* Determined by the Hatfield and Thirkell Method.

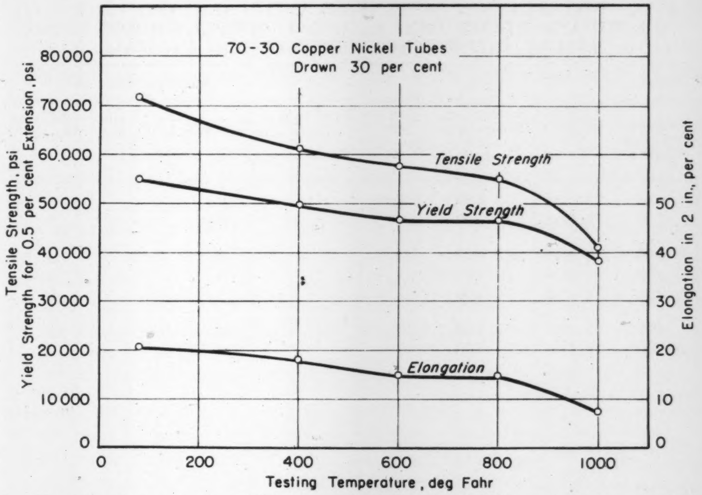


FIG. 8.—Hot Tensile Properties of 70-30 Copper-Nickel Tubes (⅝-in. outside diameter by 0.069-in. wall) Drawn 30 per cent and Relief Annealed for 1 hr at 1100 F.

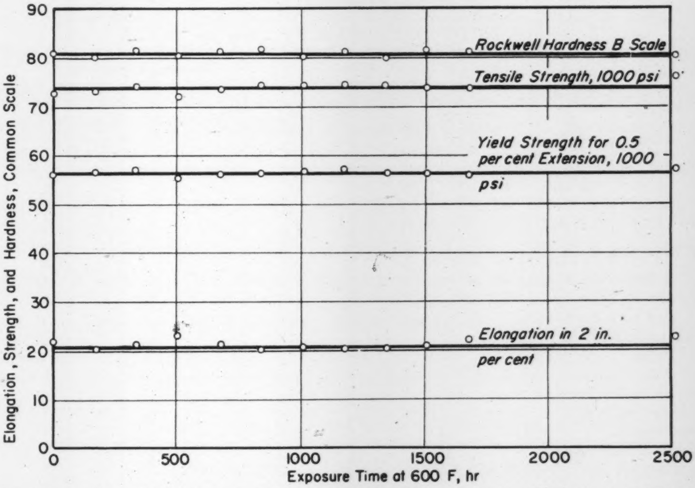


FIG. 9.—Properties of 70-30 Copper-Nickel Tubes (⅝-in. outside diameter by 0.069 in. wall) Drawn 30 per cent and Relief Annealed for 1 hr at 1100 F *versus* Exposure Time at 600 F.

the strength is still at attractively high values.

The importance or need of stress-relief annealing of this alloy with respect to the possibility of stress-corrosion cracking may be questioned, for it is one of the most resistant of copper-base materials. Regardless of this, the residual stress in

TABLE IX.—CORRELATION OF TENSILE AND U-BENDING PROPERTIES OF DRAWN AND RELIEF ANNEALED 70-30 COPPER-NICKEL TUBES 0.624 IN. OUTSIDE DIAMETER BY 0.051 IN. WALL.

Relief Anneal		Tensile Strength, psi	Yield Strength (0.5 per cent Elongation under Load), psi	Elongation in 2 in., per cent	Bending Conditions		
Time, hr	Temperature, deg Fahr				Bend Radius, in. .... 1 in. Center-to-Center Distance,.... 2 in. Ratio: $\frac{\text{Bend Radius}}{\text{OD}}$ 1.6	1⅛ 2¼ in. 1.8	1¼ 2½ in. 2.0
1.....	750	78 700	73 400	14.5[a]	*B*	*B*	*B*
1.....	750	79 300	71 500	16[b]	*B*	*B*	*A*
1.....	950	73 500	63 600	20[a]	*B*	*A*	*A*
1.....	950	74 800	63 700	18.5[b]	*B*	*A*	*A*
1.....	950	78 400	70 800	17.5[c]		*B*	*A*
1.....	1100	69 200	53 500	26[a]	*A*	*A*	*A*
1.....	1100	70 700	54 700	26[b]	*A*	*A*	*A*

Grain size prior to final draw.. 0.025 mm
Final reduction.. 28 per cent

A = Successful bends.
B = Bend failures.
[a] As annealed.
[b] Roll straightened.
[c] Straightened on both roll and tandem inclined roll straighteners.

TABLE X.—CORRELATION OF TENSILE AND U-BENDING PROPERTIES OF DRAWN AND RELIEF ANNEALED 70-30 COPPER-NICKEL TUBES 0.625 IN. OUTSIDE DIAMETER BY 0.064 IN. WALL.

Relief Anneal		Tensile Strength, psi	Yield Strength (0.5 per cent Elongation under Load), psi	Elongation in 2 in., per cent	Bending Conditions		
Time, hr	Temperature, deg Fahr				Bend Radius, in. .... 1 in. Center-to-Center Distance, in.. 2 in. Ratio: $\frac{\text{Bend Radius}}{\text{OD}}$ 1.6	1⅛ 2¼ 1.8	1¼ 2½ 2.0
1.....	950	75 200	63 500	20[a]	*B*	*A*	*A*
1.....	950	73 900	61 900	19.5[b]	*B*	*B*	*A*
1.....	950	75 200	65 300	20[c]	*B*	*B*	*A*
1.....	1100	71 500	55 800	27[a]	*A*	*A*	*A*
1.....	1100	65 700	43 500	29.5[b]	*A*	*A*	*A*
1.....	1100	71 700	58 300	23[c]	*A*	*A*	*A*

Grain size prior to final reduction.. 0.030 mm
Final reduction.. 29 per cent

A = Successful bends.
B = Bend failures.
[a] As annealed.
[b] Roll straightened.
[c] Straightened on both roll and tandem inclined roll straighteners.

the tube as drawn 40 per cent and as subsequently annealed for 1 hr at temperatures as high as 1100 F is shown in Fig. 7. Residual stress was measured by the Hatfield-Thirkell[3] procedure as applied to tube specimens slit along one element. Some relief of stress is attained at 600 F, and complete relief is realized at 1000 F. This contradicts some technical literature which cites 475 F as a proper relief annealing temperature for this alloy.

---

[3] Hatfield and Thirkell, "Season Cracking," *Journal of the Institute of Metals*, Vol. 22, p. 67 (1919).

Tensile properties from room temperature to 1000 F are shown in Fig. 8 for the $\frac{5}{8}$-in. outside diameter by 0.069-in. wall tube as drawn 30 per cent and relief annealed at 1100 F for 1 hr. The drop in strength and elongation is gradual until 1000 F is reached. At that temperature the tensile fractures are brittle for the first time. At and below 800 F the fractures are ductile and indicative of a tough material.

The stability of the relief annealed condition for prolonged exposure at elevated temperature is of interest. We have accordingly exposed samples of the same $\frac{5}{8}$-in. outside diameter by 0.069-in. tube, drawn 30 per cent and relief annealed at 1100 F, for periods as long as 2500 hr at 600 F, a temperature which is pertinent to the operation of some feedwater heaters. Subsequent hardness and tensile properties at room temperature are shown in Fig. 9. There is no change in properties and the material appears stable.

U-bending experiments have been conducted on commercial equipment with tube $\frac{5}{8}$-in. outside diameter by 0.051-in. wall as drawn 28 per cent, relief annealed and straightened and with tubes of $\frac{5}{8}$-in. outside diameter by 0.064-in. wall as drawn 29 per cent, relief annealed and straightened. Tubes of these diameters, with the same or heavier wall thicknesses, are of interest for service in feedwater heaters. The ability of a given tube to take a bend successfully is directly related to its elongation in the tension test, as is evident by inspection of Tables IX and X. The center-to-center distances for these bends are such as might be used for the innermost course of a bent-tube unit. Bending naturally becomes less difficult in the succeeding courses where the center-to-center distances are larger.

MR. B. J. SIROIS (*author*).—I want to thank Messrs. Burghoff, Kuzmenko, and Nole for reporting data which confirm certain preliminary and exploratory work done by us prior to the more extensive investigation by Battelle Memorial Institute. The work done at Battelle Memorial Institute produced the data needed to support our request to the ASME Boiler and Pressure Vessel Code for improved design stress values which would permit the alloy to be used in feedwater heaters intended to operate at high temperatures and pressures.

We agree that a ductility measured as an elongation of the order of 25 per cent in 2 in. is needed to make U-bends on a $\frac{5}{8}$-in. outside diameter heat exchanger tube to a radius of 1 in. However, manufacturers of feedwater heaters, realizing the economies resulting from using tubes with the improved mechanical properties disclosed in this paper, have indicated a willingness to sacrifice some ductility in the tube in order to obtain these improved properties and also to relocate the inner row of tubes in feedwater heaters to permit tubes U-bent to radii of the order of 2 to 2.2 times the tube diameter.

MECHANICAL PROPERTIES OF A HIGH-PURITY LEAD AND A 0.058 PER CENT COPPER-LEAD ALLOY AT ELEVATED TEMPERATURES*

By T. E. Tietz[1]

Synopsis

The tensile, compression, shear, bearing and creep properties of a high-purity lead and a 0.058 per cent copper-lead alloy were evaluated at test temperatures of 100, 175, 250, and 325 F.

Tensile properties evaluated included the stress-strain curve to fracture, the ultimate strength, elongation, modulus of elasticity, proportional limit, and yield strength. Compression properties evaluated were the modulus of elasticity, proportional limit, yield strength, and stress-strain curve to 5 per cent strain. The ultimate shear strength and the bearing yield strength and ultimate bearing strength were determined. Creep stress - time curves were obtained for total strain values of 0.2, 0.5, 1.0, and 2.0 per cent, for creep times of from 1 to 500 hr.

The properties of lead make it the most practical shielding material presently available. In the design and development of nuclear-powered aircraft, the structural problem of providing effective over-all shielding with the lowest possible weight is most important and requires that the mechanical properties of lead be sufficiently known. However, because of its low strength even at room temperature, lead has not been seriously considered as a structural material and its mechanical properties have not been adequately determined. In addition, the few studies which have been conducted on the mechanical properties of lead have been at or near room temperature.

The objective of this program was to determine the tensile, compression, shear, bearing, and creep properties of a commercially pure lead and a lead alloy, at four test temperatures up to 325 F.

Test Material

The two materials evaluated in this program were a high-purity lead (Doe Run Brand refined lead, 99.995 per cent lead) and a copperized lead (Copperized Doe Run Brand refined lead, 0.058 per cent copper). The eutectic point in the lead-copper system occurs at approximately 0.06 per cent copper. The chemical composition of the two test materials is given in Table I. In the case of the copperized lead, bars extruded from lot I were used for the tension, shear, and creep specimens, and from lot II for the compression and bearing specimens. The analyses are, for all practical purposes, identical for the two lots of copperized lead.

The lead was extruded into bar forms having the following cross-sections:

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Research Scientist, Metallurgy and Ceramics Laboratories, Lockheed Missiles and Space Division, Palo Alto, Calif. Formerly Senior Metallurgist, Stanford Research Institute, Menlo Park, Calif.

Type of Test	Extruded Cross-Section
Tension and creep	½ × 1¼ in.
Compression	½ × ½ in.
Shear	½ in. round
Bearing	½ × 4 in.

The extrusion slugs were cast at 900 to 950 F. The slugs were 12 in. long by 4½ in. in diameter, after cropping, with the exception of the single slug from which the bearing specimens were extruded using a different extrusion press. The extrusion was done at room temperature.

TEST SPECIMENS

The dimensions of the four types of test specimens used in this program are given in Fig. 1. All test specimens were machined with their axes parallel to the extrusion direction.

All test specimens were given an annealing treatment of 2 hr in an oven at 400 F after machining and prior to testing, in order to remove any cold-work within the specimens due to machining and handling. The grain sizes of the two materials were considerably different, both in the as-received and in the annealed conditions; the copper addition acted as a grain size refiner. The average annealed grain size of the high-purity lead was about 0.55 grains per mm and that of the copperized lead about 18 grains per mm.

EXPERIMENTAL APPARATUS AND PROCEDURES

The tension, compression, shear, and bearing tests were conducted with a Baldwin Universal hydraulic testing machine, using a 600-lb full-scale load range. This machine was equipped with a forced air convection furnace. A dial gage mounted on the movable crosshead was used to measure and control the crosshead travel rate during testing, and a Baldwin deflectometer was used to record the crosshead travel for tests in which crosshead travel was used as the strain measurement. The specimen was placed in the furnace approximately 1 hr before loading to permit the specimen to attain the test temperature. A thermocouple was attached directly to the test specimen during each test, and the test temperatures were maintained within ±2 F.

Tension Tests:

The tension test specimens had a reduced cross-section of ½ by ½ by 3 in. A differential transformer type extensometer with a 2-in. gage length was used to evaluate the elastic properties in tension and the tensile stress-strain curve to 2 per cent strain, at a crosshead rate of 0.015 in. per min for a strain rate of approximately 0.005 per min.

TABLE I.—CHEMICAL COMPOSITION OF TEST MATERIALS, PER CENT.

Element	High-Purity Lead	Copperized Lead	
		Lot I	Lot II
Lead	99.9951	99.9398	99.9404
Copper	0.0001	0.0577	0.0577
Arsenic, antimony, tin	0.0004	0.0006	0.0004
Iron	0.0001	0.0001	0.0001
Zinc	0.0002	0.0001	0.0001
Cadmium	0.0029	0.0006	0.0005
Nickel, cobalt	0.0000	0.0000	0.0000
Bismuth	0.0006	0.0007	0.0005
Silver	0.0006	0.0004	0.0003

For determining the entire stress-strain curve, the strain was determined by using the deflectometer to measure crosshead travel, and in this case the entire 3-in reduced section served as the gage length. For these tests, the crosshead rate was maintained at 0.150 in. per min for a strain rate of approximately 0.05 per min.

In both cases the stress-strain curves were recorded autographically.

Compression Tests:

The compression tests were conducted on ½ by ½ by 2-in. specimens, at a cross-

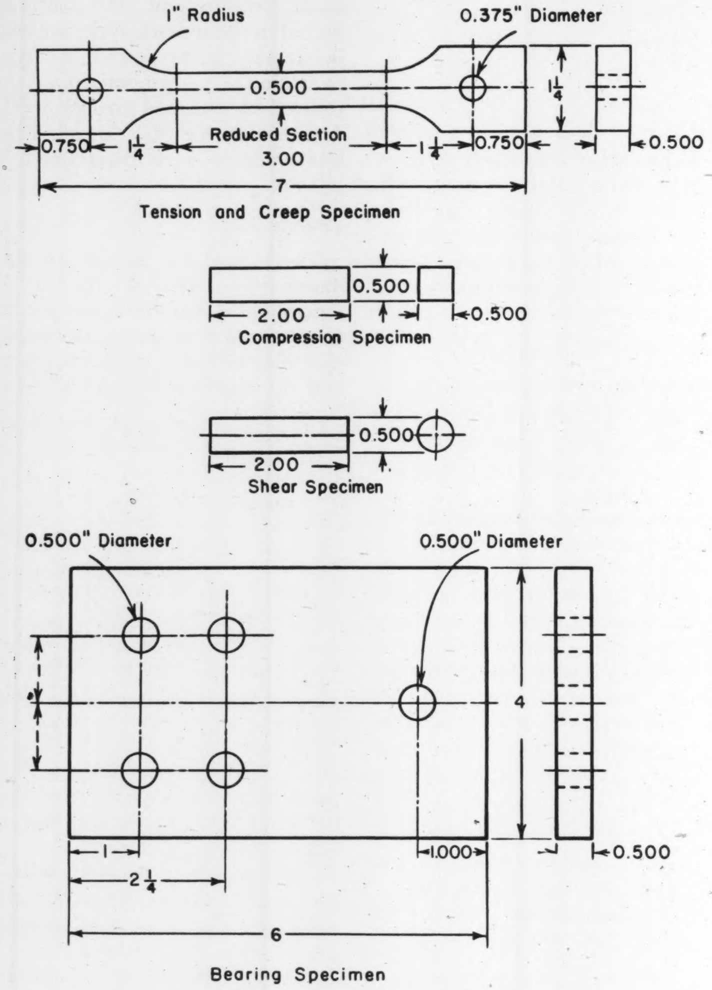

1" Radius
0.375" Diameter
0.500
1¼
Reduced Section
3.00
0.750
1¼
1¼
0.750
0.500
7
Tension and Creep Specimen
0.500
2.00
0.500
Compression Specimen
0.500
2.00
Shear Specimen
0.500" Diameter
0.500" Diameter
4
1
2¼
1.000
0.500
6
Bearing Specimen

FIG. 1.—Dimensions of Test Specimens.

head travel rate of 0.010 in. per min, for a strain rate of 0.005 per min. A differential transformer type extensometer with a 1-in. gage length was used to evaluate the elastic properties in compression and the compression stress-strain curve to 5 per cent strain.

Initial compression tests were conducted with a compression subpress. At the elevated test temperatures, difficulty was encountered as a result of binding of the press. For this reason, all the compression tests were conducted without a subpress, using flat-ground bearing plates placed on top and bottom of the specimen during the test.

Shear Tests:

The shear test specimens, $\frac{1}{2}$ in. in diameter by 2 in. long, were tested[3] in double shear, using a constant crosshead travel rate of 0.005 in. per min.

TABLE II.—TENSION TEST RESULTS.

Ultimate tensile strength and elongation at a strain rate of 0.05 per min

Test Material	Test Temperature, deg Fahr							
	100		175		250		325	
	Ultimate Tensile Strength, psi	Elongation, per cent	Ultimate Tensile Strength, psi	Elongation, per cent	Ultimate Tensile Strength, psi	Elongation, per cent	Ultimate Tensile Strength, psi	Elongation, per cent
High-purity lead 1......	1828	46.0	1240	57.3	788	64.0	498	77.6
2......	1920	44.6	1196	42.6	798	50.6	488	72.0
3......	1852	40.0	1204	36.0	768	68.3	492	83.3
Average..........	1867	43.5	1213	45.3	785	61.0	493	77.6
Copperized lead 1......	1580	53.6	1164	46.0	826	42.6	636	46.6
2......	1604	46.3	1148	47.0	846	50.6	638	56.0
3......	1570	53.0	1162	47.7	844	48.3	642	42.7
Average..........	1585	51.0	1158	46.9	839	47.2	639	48.4

Bearing Tests:

The bearing test specimen consisted of a flat 4 by 6 by $\frac{1}{2}$-in. plate with a $\frac{1}{2}$-in. diameter bearing hole whose center was 1 in. from the edge of the plate. A $\frac{1}{2}$-in. steel pin was inserted in this hole and pulled at a constant crosshead rate of 0.005 in. per min.

The yield strength, defined as 2 per cent offset of the hole diameter, was determined by means of a small dial gage, which measured the relative pin movement and was mounted directly on the bearing test fixture. The stress-deformation curve to fracture was determined by measuring the crosshead movement and recording the load and deformation autographically.

Creep Tests:

Equipment.—One creep unit was used for each test temperature. The furnaces for maintaining constant test temperatures consisted of cylindrical tanks 10 in. in diameter and 24 in. long, with a vertical, 2-in. inside diameter tube passing through the center of each tank. The specimens were heated and tested in an air atmosphere within the tubes. Suitable packing was used at the tube ends to prevent air convection through the tubes.

The specimens were loaded through 5 to 1 lever-arm systems in the case of the lower two test temperatures and by dead-weight loading in the case of the upper two temperatures. The test temperatures of 100 and 175 F were maintained constant within ±2 F by water and thermostatic control; the test temperatures of 250 and 325 F were maintained constant within ±1 F by boiling glycol-water solutions and condenser systems.

Conventional type creep extensometers were used with two pairs of gage blocks which were attached to the specimen to give a 2-in. gage length by means of four hardened, conical points pressed into the specimen and held in place by coil springs. The relative movement of the gage blocks was transferred through two pairs of extension rods to a 0.0001-in. least-count dial gage outside the furnace. Upper guide blocks were used which had polished surfaces that were free to move along the axis of the polished pulling bar.

Test Procedure.—The test specimen was carefully assembled, using a special mounting board for aligning the pulling bars and extensometer. A thermocouple for temperature checks during test was tied directly to the specimen at the center of the 2-in. gage length.

A set of chromel-alumel thermocouples were calibrated, using a boiling distilled water bath which gave a calibration point midway between the four test temperatures. Four thermocouples were selected for uniformity, and one thermocouple was used for all creep tests at one test temperature.

The specimen assembly was inserted into the furnace two hours prior to application of the load in order to permit the specimen to attain the test temperature. The load was then gradually applied by hand over a period of about $\frac{1}{2}$ min; zero time was taken when the full load was on the specimen.

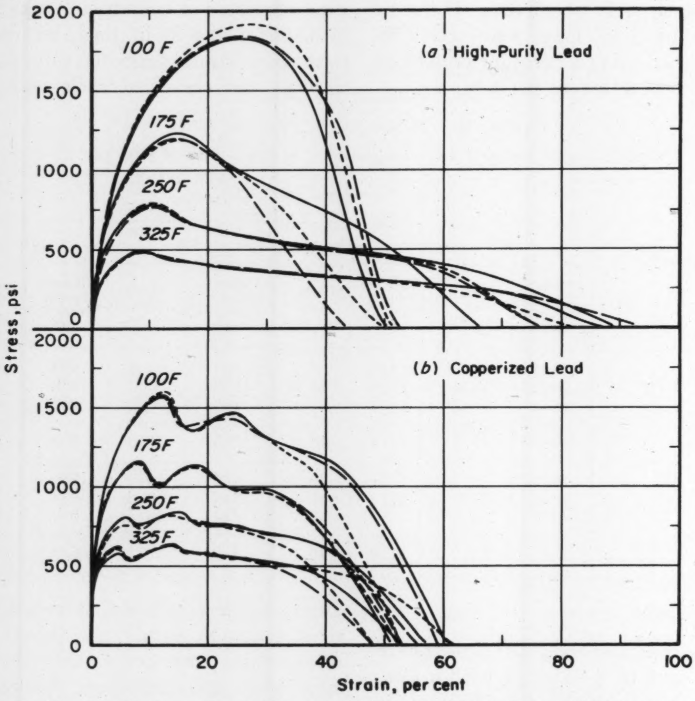


FIG. 2.—Tensile Stress-Strain Curves to Failure at a Strain Rate of 0.05 per min.

EXPERIMENTAL RESULTS

Tension Tests:

Stress-Strain Curves to Failure.—The stress-strain curves to failure are presented in Fig. 2 for the two test materials. The tests were conducted in triplicate at each of the test temperatures: 100, 175, 250, and 325 F, and at a strain rate of 0.05 per min. Data on the ultimate tensile strength and elongation are summarized in Table II. The elongation values given in the table were obtained by using gage marks originally 3.00 in. apart on the specimen.

The triplicate tests under the same test conditions gave very similar stress-strain curves up to the point of necking. Beyond this point the curves differed considerably, due to differences in necking conditions, probably influenced by variations in grain size and grain orientation within the different specimens. The fracture strength was very nearly zero for all tests, as is indicated by the curves on Fig. 2.

The stress-strain curves for the high-purity lead were quite typical, each exhibiting one maximum, whereas the curves for the copperized lead all had two or more maxima. In the latter case, necking did not occur at the first maximum in the stress-strain curves; this effect was possibly associated with recrystallization of the finer-grain-size copperized lead during test.

The total elongation values shown in Fig. 2 include some elongation of the specimen bearing holes, which in extreme cases amounted to about $\frac{1}{16}$ in. per hole, whereas in most cases it was less than $\frac{1}{32}$ in. per hole. Some elongation also occurred in the fillet areas outside the 3-in. reduced section, with the result that the indicated strain values are larger than actual. This problem was not encountered in the other tension or compression tests, as the strain measurements were made directly on the specimen gage length.

Figure 3 presents the average values of the ultimate tensile strength and elongation as a function of test temperature. The high-purity lead had a higher tensile strength at 100 and 175 F than the copperized lead and a lower value at the higher test temperatures of 250 and 325 F. It should be noted in Fig. 2 that at any one test temperature the rate of strain hardening of the copperized lead was greater than that for the high-purity lead almost up to the point of the first maximum. If only the maximum associated with necking had occurred, these tests undoubtedly would have exhibited higher ultimate tensile strength values.

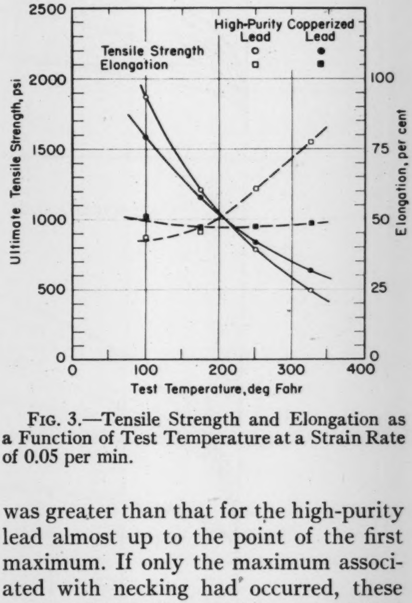


FIG. 3.—Tensile Strength and Elongation as a Function of Test Temperature at a Strain Rate of 0.05 per min.

The elongation for the copperized lead was approximately constant at about 50 per cent at all test temperatures, whereas the average elongation of the high-purity lead increased from 43 to 77 per cent over the temperature range from 100 to 325 F.

Elastic Properties.—A second set of 24 tension specimens was used to evaluate the elastic properties and the stress-strain curve to 2 per cent strain. A cross-head travel rate of 0.015 in. per min was used for these tests, for a strain rate of 0.005 per min. The results of these tests are shown in Fig. 4 for the two test materials tested at the four test temperatures. The triplicate tests at any one temperature are in good agreement. In order to show more clearly the effect of temperature, the data of Fig. 4 were plotted as stress *versus* test temperature for constant values of total strain and are presented in Fig. 5.

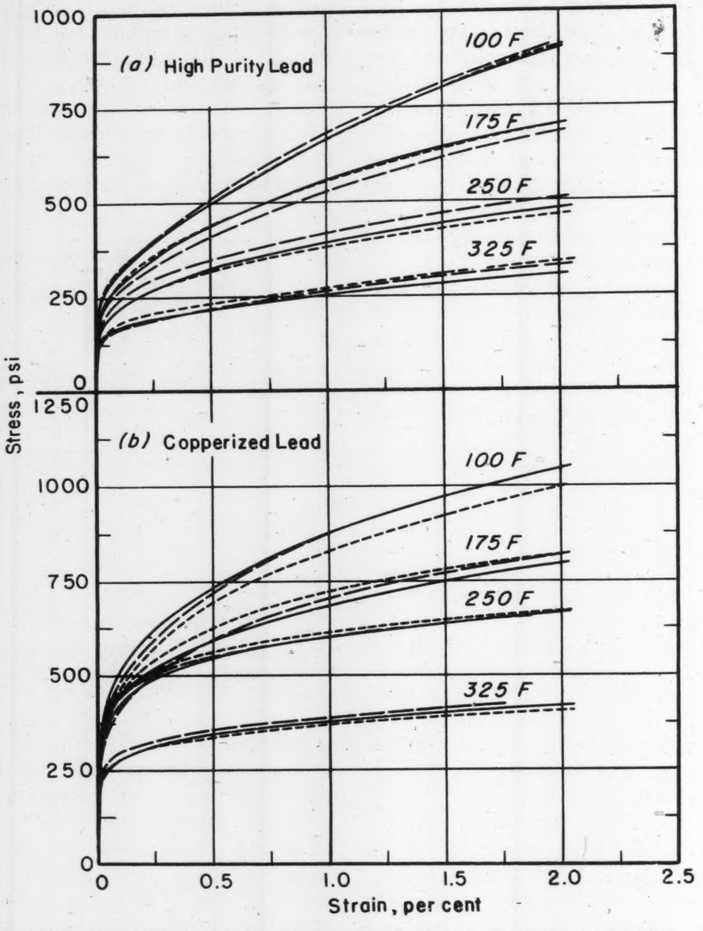


FIG. 4.—Tensile Stress-Strain Curves to 2 per cent Strain at a Strain Rate of 0.005 per min.

The elastic properties were evaluated from the recorded load-deformation charts by initially using a high strain magnification to a strain of 0.3 per cent, 1 in. of chart being equal to 0.05 per cent strain. The test was then completed by using a low magnification, 1 in. of chart equal to 0.2 per cent strain, to a total strain of 2.0 per cent. At this point the specimen was unloaded and reloaded three times, using the high strain magnification, the loading curves being used to evaluate the elastic modulus. This procedure was used to evaluate the modulus because the initial yield point was too low in most cases to make evaluation possible.

Tension data for the modulus of elasticity, proportional limit, and yield strength are summarized in Table III. The tabulated values of the modulus of elasticity were calculated on the basis of the reduced cross-sectional area at 2 per cent strain. For the high-purity lead, the average value of the modulus was 2.7×10^6 psi, with no apparent decrease from 100 to 250 F. Values at 325 F could not be evaluated with sufficient accuracy. The proportional limit and yield strength both show a decrease of about 50 per cent with test temperature from 100 to 325 F. For the copperized lead, the average value of the modulus was 2.2×10^6 psi with about a 10 per cent decrease at 325 F; the proportional limit and yield strength remained practically constant from 100 to 250 F and then decreased about 35 per cent at 325 F.

Effect of Strain Rate.—The effect of strain rate on the stress-strain curve was evaluated at 100 and 250 F for both test materials. Figure 6 presents tests conducted at a strain rate of 0.005 per min, along with previous tests conducted at a rate of 0.05 per min. For the high-purity lead, a decrease in the strain rate from 0.05 to 0.005 per min resulted in a decrease in the ultimate tensile strength of about 30 per cent at both test temperatures and also in a decrease in strain at the ultimate and in the total strain to fracture. For the copperized lead, the same decrease in strain rate resulted in a 15 per cent decrease in the ultimate and a slight decrease in the elongation to fracture.

Compression Tests:

Stress-strain curves in compression to 5 per cent strain are given in Fig. 7. Triplicate tests were conducted at each test temperature for both materials, using a crosshead travel rate of 0.010 in. per min for a strain rate of 0.005 per min. There is some overlapping in the curves for the high-purity lead at small values of strain for the different test temperatures. This is most likely due to small sampling differences which initially existed from specimen to specimen. After 1 per cent strain, the curves at any one test temperature are in close agreement. For the high-purity lead at 250 and 325 F, in some cases a decrease in the stress-

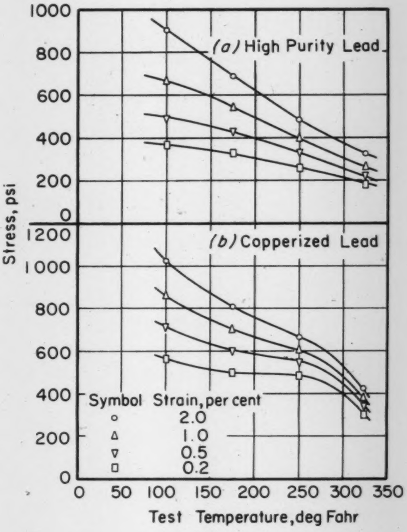


FIG. 5.—Tensile Flow Stress *versus* Test Temperature for Given Values of Strain at a Strain Rate of 0.005 per min.

TABLE III.—TENSION DATA—MODULUS OF ELASTICITY, PROPORTIONAL LIMIT, AND YIELD STRENGTH.

At a strain rate of 0.005 per min

	Test Temperature, deg Fahr	Modulus of Elasticity, 10^6 psi				Proportional Limit, psi	Yield Strength (0.2 per cent offset), psi
		E_1	E_2	E_3	E_{avg}		
High-purity lead.....	100	3.0	3.0	3.1	3.0	190	372
	100	2.6	2.6	2.7	2.6	170	380
	100	2.6	2.6	2.6	2.6	180	384
	Average				2.7	180	379
	175	...	...	...	...	150	332
	175	2.6	2.6	2.5	2.6	150	312
	175	2.7	2.5	2.5	2.6	...	...
	175	2.6	2.6	2.6	2.6	103	347
	Average				2.6	134	330
	250	2.4	2.9	2.4	2.6	130	260
	250	2.2	2.5	2.6	2.4	140	288
	250	3.1	2.8	3.0	3.0	130	260
	Average				2.7	133	269
	325	...	...	...	...	90	188
	325	...	...	...	...	72	180
	325	...	...	...	...	90	200
	Average					84	189
Copperized lead.....	100	2.5	2.5	2.4	2.5	328	612
	100	2.2	2.2	2.4	2.3	200	580
	100	2.3	2.2	2.1	2.2	300	560
	Average				2.3	276	584
	175	1.9	2.4	2.0	2.1	350	512
	175	2.2	2.2	2.3	2.2	260	488
	175	2.4	2.3	...	2.3	270	528
	Average				2.2	293	509
	250	...	2.3	2.3	2.3	260	488
	250	1.8	1.8	1.9	1.8	260	500
	250	2.9	2.6	...	2.7	312	504
	Average				2.3	277	498
	325	2.1	2.1	2.0	2.1	200	308
	325	...	...	...	...	208	320
	325	2.0	2.0	1.9	2.0	160	304
	Average				2.0	189	311

strain curve occurred after 3 per cent strain. In these cases no unusual condition was observed during test; a possible explanation is that recrystallization took place at these higher test temperatures under the particular test conditions.

The compression data are shown in Fig. 8 as stress *versus* test temperature for various values of total strain. At the lower test temperatures the stress values for the copperized lead are only slightly higher than those for the high-purity lead. At 325 F this difference becomes larger, the curves for the high-purity lead decreasing more rapidly above 250 F than those for the copperized lead.

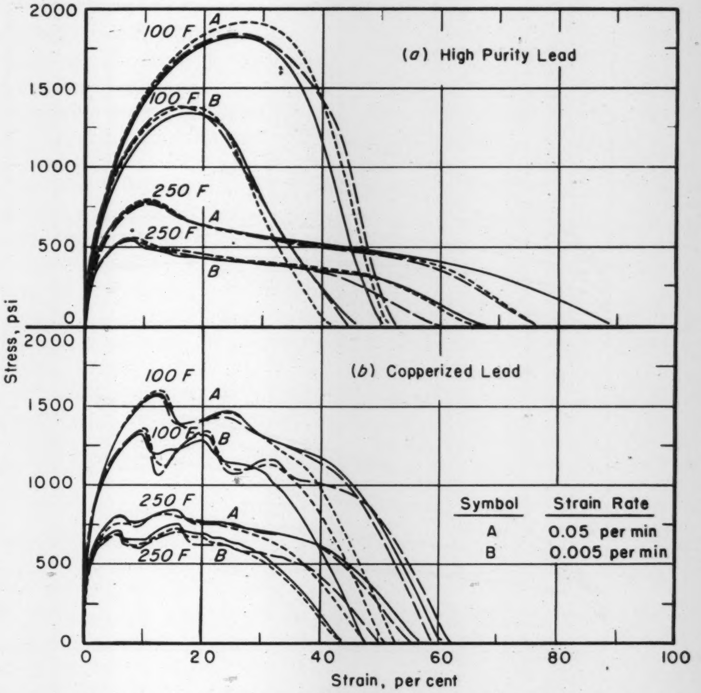


FIG. 6.—Effect of Strain Rate on the Tensile Stress-Strain Curves at 100 and 250 F.

The elastic properties in compression were evaluated from the recorded load deformation charts in a manner similar to that used for the tension tests. A high strain magnification—1 in. of chart equal to 0.10 per cent strain—was used to a strain of 0.3 per cent. The test was completed at a low magnification—1 in. of chart equal to 0.4 per cent strain—to a strain of 5 per cent. At this point, the specimen was unloaded and reloaded three times, using the high strain magnification, the loading curves being used to evaluate the elastic modulus.

The modulus of elasticity, proportional limit, and yield strength data in compression are summarized in Table IV. The tabulated values of the modulus of elasticity were calculated on the basis of the increased cross-sectional area at 5 per cent strain.

The average value of the modulus was 2.6×10^6 psi for both materials, and decreased from 2.8×10^6 psi at 100 F to about 2.4×10^6 psi at 325 F. The proportional limit and yield strength values were about the same for the two leads at each of the three lower test temperatures; at 325 F the values for the copperized lead were about 50 per cent higher than those for the high-purity lead.

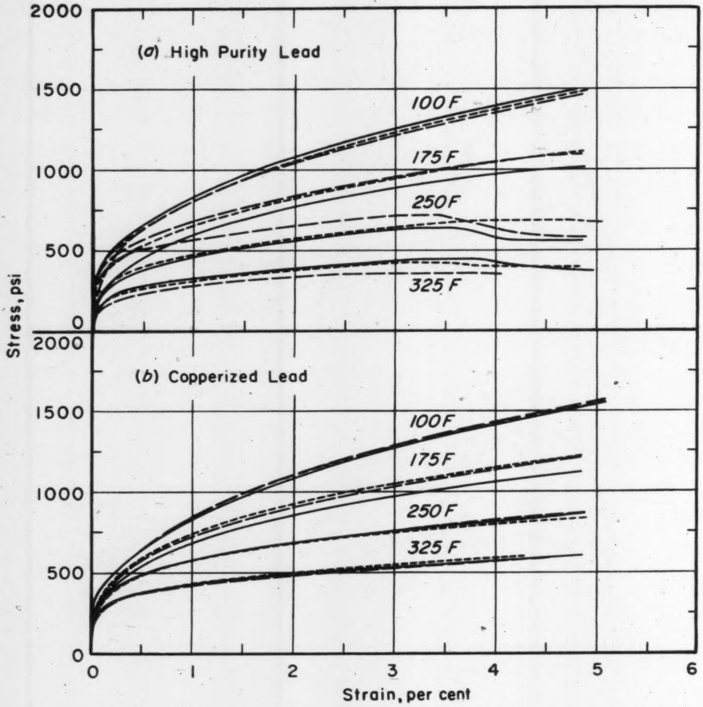


FIG. 7.—Compression Stress-Strain Curves to 5 per cent Strain at a Strain Rate of 0.005 per min.

Shear Tests:

The shear test results are summarized in Fig. 9 as ultimate shear strength *versus* test temperature. These tests were conducted by testing pins $\frac{1}{2}$ in. in diameter by 2 in. long in double shear, using a constant crosshead travel rate of 0.005 in. per min.

The data points in Fig. 9 represent the average values for each set of triplicate tests. At 100 F the ultimate shear strength of the high-purity lead was about 20 per cent higher than that for the lead containing 0.058 per cent copper. However, the shear strength of the high-purity lead decreased more rapidly with test temperature than that of the copperized lead and was lower at temperatures above 160 F.

The shear tests on the copperized lead behaved in a similar manner to the tension tests on the copperized lead, in that more than one maximum in the load occurred during each test.

Bearing Tests:

The bearing tests were conducted at a crosshead travel rate of 0.005 in. per min, and the stress-deformation curves to failure are given in Fig. 10. For this purpose the stress is defined as the load divided by the bearing area, which is taken as the diameter of the bearing hole times the bearing plate thickness.

The bearing yield strength, defined as 2 per cent offset of the hole diameter, and ultimate bearing strength are presented in Fig. 11 as a function of temperature. The bearing yield strength for the copperized lead is higher than that for the high-purity lead at all test temperatures. The ultimate bearing strength for the high-purity lead at 100 F is higher than that for the copperized, but becomes lower at temperatures above about 140 F. Inspection of the stress-deformation curves in Fig. 10 for the copperized lead shows that they exhibit a definite decrease in the rate of strain hardening, then an increase prior to the ultimate.

Creep Tests:

The original creep data are presented as total strain *versus* creep time in Fig. 12 for the high-purity lead and in Fig. 13 for the copperized lead.

Some crossing of the strain-time curves occurred at very low strain values of less than 0.0004. No fundamental significance is given to this behavior which was probably due to one or a combination of the following factors: sampling differences, slight variations in loading conditions, and possible initial lag in extensometer movement.

Greenwood and Worner,[2] studying the creep of lead, demonstrated that a rapid increase in the creep rate occurred when recrystallization took place during test. The longer time and higher temperature creep tests of the current study were plotted on regular coordinate paper as strain against time. All these curves showed a continually decreasing creep rate with time, indicating that no recrystallization took place under the given test conditions. This was not confirmed, however, by microstructure studies.

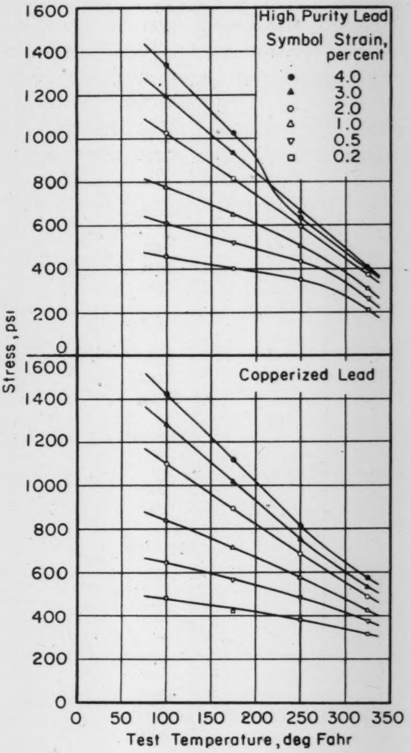


FIG. 8.—Compression Flow Stress *versus* Test Temperature for Given Values of Strain at a Strain Rate of 0.005 per min.

In Figs. 14 and 15 the creep results are

---

[2] J. Neill Greenwood and H. K. Worner, "Types of Creep Curves Obtained with Lead and Its Dilute Alloys," *Journal of the Institute of Metals*, Vol. 64, No. 1, p. 135 (1939).

TABLE IV.—COMPRESSION DATA—MODULUS OF ELASTICITY, PROPORTIONAL LIMIT, AND YIELD STRENGTH.

At a strain rate of 0.005 per min

	Test Temperature, deg Fahr	Modulus of Elasticity, 10^6 psi				Proportional Limit, psi	Yield Strength (0.2 per cent offset), psi
		E_1	E_2	E_3	E_{avg}		
High-purity lead.....	100	2.8	2.8	2.8	2.8	282	500
	100	...	2.6	2.6	2.6	81	440
	100	...	3.1	3.0	3.1	202	472
	Average				2.8	188	471
	175	...	2.5	2.7	2.6	100	328
	175	2.8	2.3	2.6	2.6	262	472
	175	2.6	2.3	2.5	2.5	202	427
	Average				2.6	188	409
	250	...	3.2	2.3	2.8	80	292
	250	2.5	...	2.3	2.4	181	440
	250	2.7	...	2.1	2.4	60	322
	Average				2.5	107	351
	325	2.5	...	...	2.5	50	228
	325	...	...	...	...	50	180
	325	...	...	...	...	80	212
	Average				2.5	60	207
Copperized lead.....	100	...	2.9	3.0	2.9	222	492
	100	2.9	2.9	...	2.9	222	492
	100	2.5	2.6	...	2.6	201	487
	Average				2.8	215	490
	175	2.8	2.8	2.6	2.7	80	408
	175	...	2.8	....	2.8	120	432
	175	3.1	3.2	3.1	3.1	120	444
	Average				2.9	107	428
	250	2.4	...	2.5	2.5	120	396
	250	...	2.6	2.6	2.6	120	396
	250	2.4	2.4	...	2.4	80	380
	Average				2.5	107	391
	325	2.4	...	...	2.4	80	320
	325	...	...	...	...	80	312
	325	...	...	...	...	120	328
	Average				2.4	93	320

summarized as stress-time curves for total strain values of 0.2, 0.5, 1.0, and 2.0 per cent for the two test materials. At 100 F the stress-time curves for the two test materials are not appreciably different, with the exception of the curves for 0.2 per cent total strain. At 175, 250 and 325 F the copperized lead was more

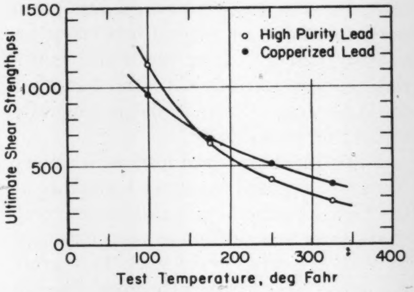


FIG. 9.—Shear Strength *versus* Test Temperature at a Cross-Head Rate of 0.005 in. per min.

creep resistant than the high-purity lead at the shorter creep times. For example, at times of about 1 hr this difference in terms of creep time to a given strain at the same stress was about 20. However, at the longer creep times the high-purity lead was more creep resistant than the copperized lead. The crossover point at which the stress-time curve for the high-purity lead became higher than that for the copperized lead occurred at shorter times as the creep temperature increased. For example, at 175 F the crossover point was 500 hr or greater; at 250 F the

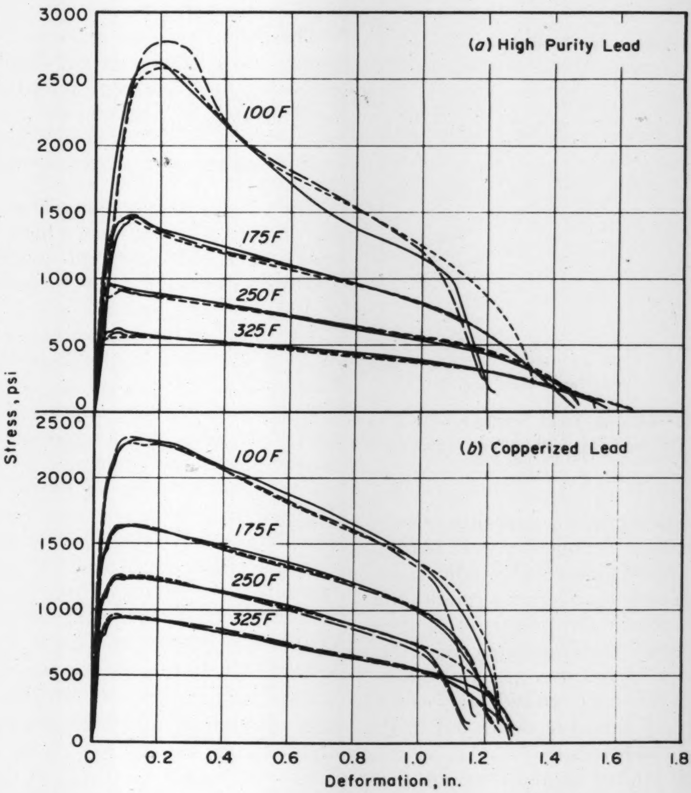


FIG. 10.—Bearing Stress-Deformation Curves to Failure at a Cross-Head Rate of 0.005 in. per min.

crossover occurred at about 60 hr for the 0.2 per cent curves and increased up to about 450 hr for the 2.0 per cent curves; at 325 F the crossover point increased from about 50 hr for the 0.2 per cent curves up to about 300 hr for the 2.0 per cent curves.

As mentioned previously, all reported results are for the test materials in the as-received condition, annealed prior to testing only to remove any work hardening due to machining and handling. The materials were tested in this condition in order to obtain the properties of these two materials as normally received. The grain sizes of the two materials as reported earlier were considerably different, both in the as-received and in the annealed conditions.

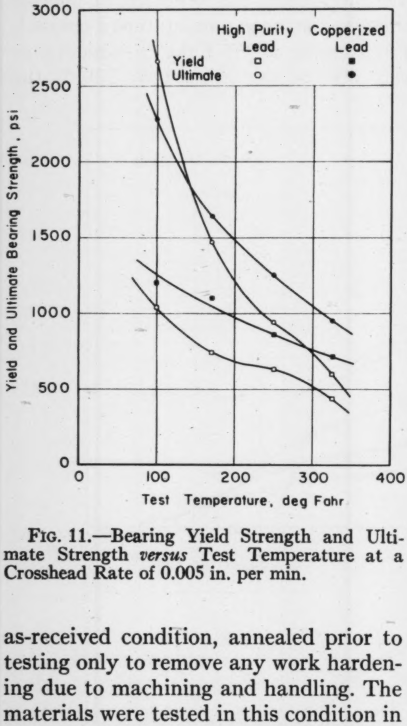


FIG. 11.—Bearing Yield Strength and Ultimate Strength *versus* Test Temperature at a Crosshead Rate of 0.005 in. per min.

Several studies on the effect of grain size on the creep of metals have shown that, for any one material, a fine grain size material is in general less creep resistant than a coarser grain size material. This is attributed to the fact that the finer grain size material is generally less stable structurally.

Thus, the copperized lead with a very fine grain size had less creep resistance at the higher temperatures and longer creep times than it would have been expected to with a coarse grain size. This grain size effect was apparently great enough to more than balance the alloy strengthening effect of the copper addition in the case of the longer creep times at the higher test temperatures.

The grain size is an important factor in the creep of metals and should be evaluated for the creep of lead at the contemplated service temperatures.

SUMMARY AND CONCLUSIONS

The tensile, compression, shear, bearing, and creep properties of a high-purity lead and a 0.058 per cent copper-lead alloy were evaluated at test temperatures of 100, 175, 250, and 325 F.

The data are summarized in graphical and tabular form in the Experimental Results section of this paper.

The reported results indicate that the rate of straining has an important effect on the mechanical properties of lead in the temperature range of 100 to 325 F. The results also suggest that the grain size has an appreciable effect on the mechanical properties of lead in the temperature range studied. Thus, when lead is to serve as a stress bearing member the factors which affect the grain size and general structural stability, such as extrusion temperature and extrusion rate, casting or rolling conditions, and possible annealing treatments, need to be evaluated with regard to the particular application.

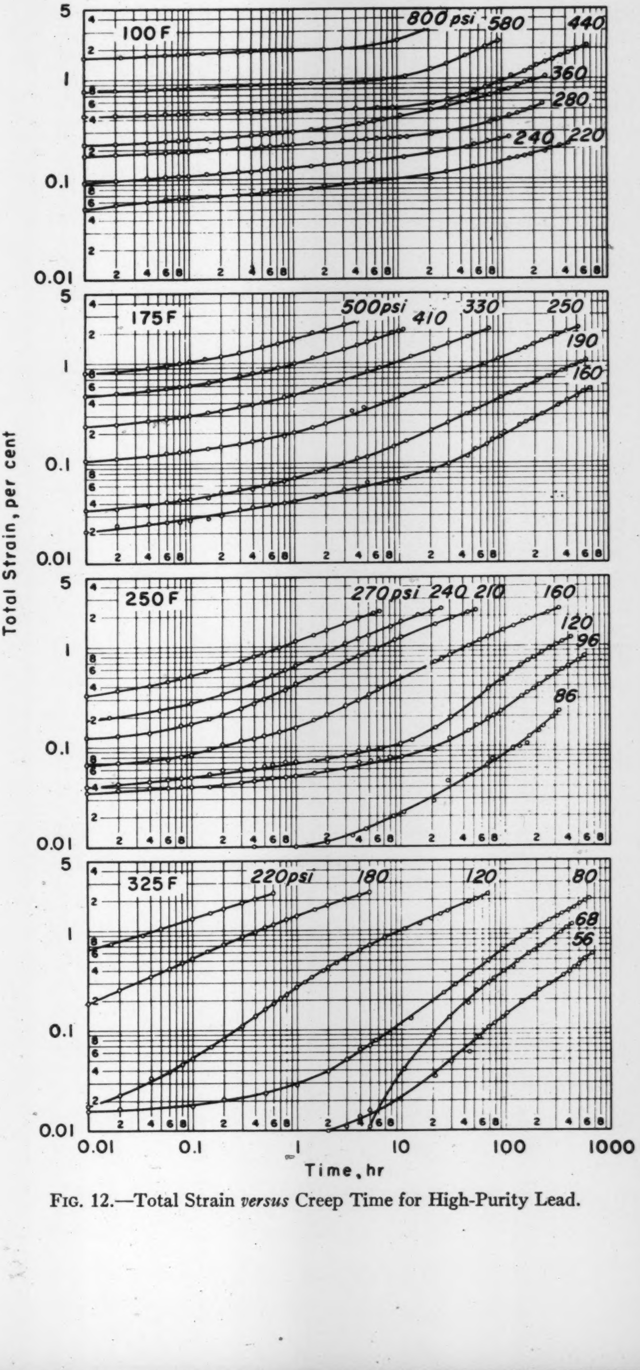


FIG. 12.—Total Strain *versus* Creep Time for High-Purity Lead.

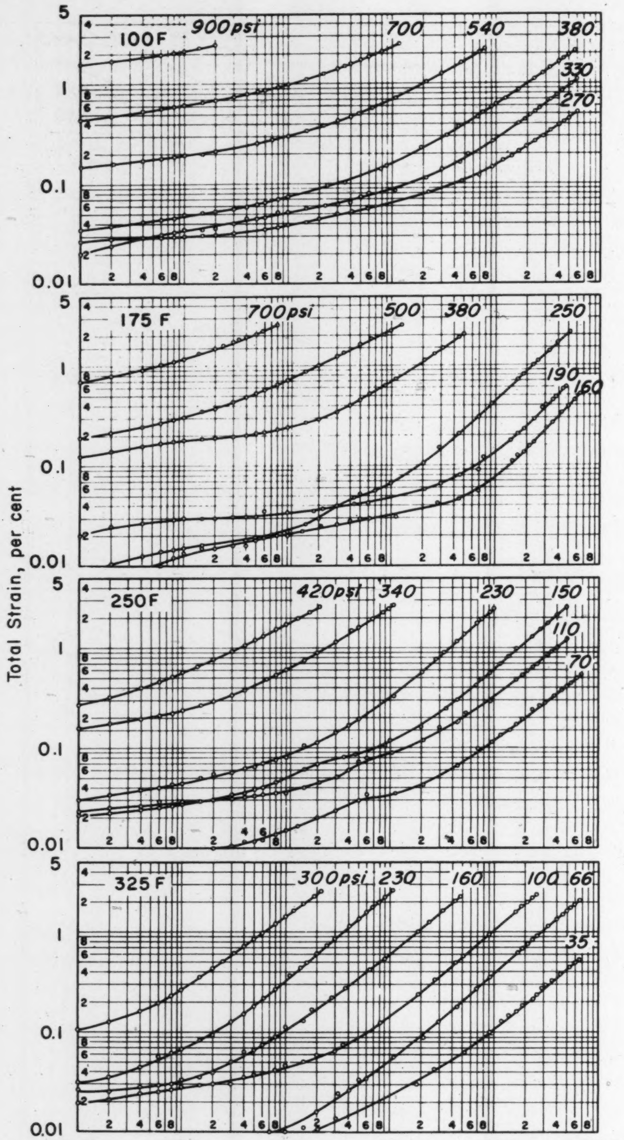


FIG. 13.—Total Strain *versus* Creep Time for Copperized Lead.

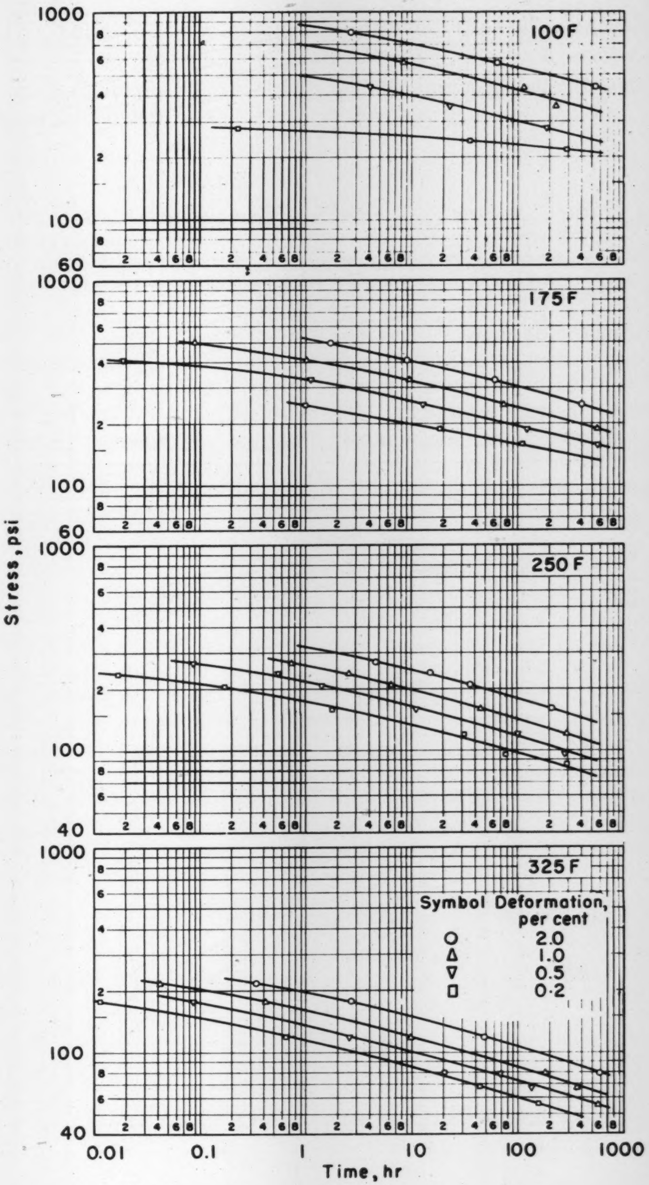


FIG. 14.—Stress *versus* Creep Time at Constant Strain Values of 0.2, 0.5, 1.0, and 2.0 per cent for High-Purity Lead.

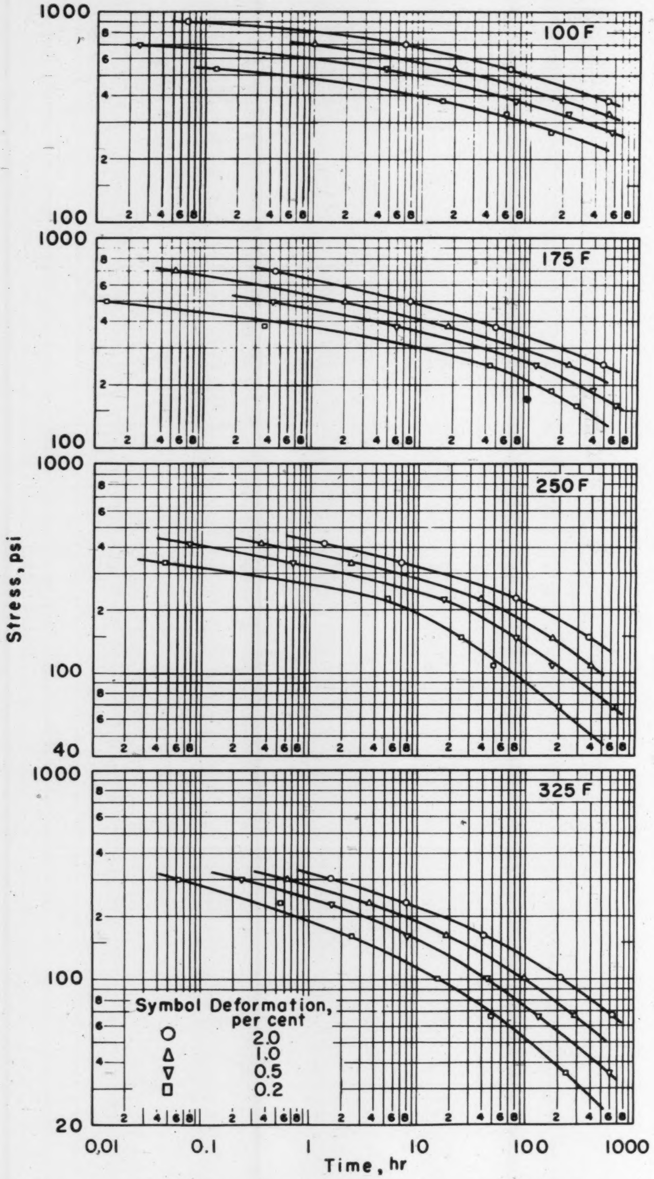


FIG. 15.—Stress *versus* Creep Time at Constant Strain Values of 0.2, 0.5, 1.0, and 2.0 per cent for Copperized Lead.

Acknowledgment:

This work was supported by the Materials Laboratory of the Wright Air Development Center under Contract No. AF 33(616)-3785. Support of this work and permission to publish this paper are gratefully acknowledged. Appreciation is expressed to Mr. A. Ruotola for his assistance throughout the testing phase of this program.

DISCUSSION

MR. R. I. JAFFEE.[1]—Copperized lead has a second phase which is substantially pure copper in small globules generally distributed through the lead, which should be strengthening as far as creep is concerned. However, it had a much finer grain size, which would be weakening so far as creep is concerned.

Would the author care to say, on basis of his work, if he had tested both materials with the same grain size, which would have been the more resistant to creep.

MR. T. E. TIETZ (*author*).—A number of investigations have shown that increasing the grain size for a given material results in increased creep resistance, although evidence has been published which indicates that an optimum grain size exists for maximum creep resistance. I think that if the two test materials in the current study were treated to result in the same grain size, the copperized lead would show somewhat greater creep resistance than the high-purity lead due primarily to the strengthening effect of the dispersed second phase as there is no appreciable solid solubility of copper in lead.

MR. F. GAROFALO.[2]—One statement that the author has made seems rather surprising. He has stated that the fracture strength is zero for the material tested. Does this mean that there has been a softening effect such as recrystallization, or is it that the load is approaching zero as the area is also approaching zero at fracture?

MR. TIETZ.—The area was also approaching zero at fracture.

MR. GARAFALO.—Actually then the true rupture strength is not zero but is not determinable.

MR. TIETZ.—The fracture strength is defined for engineering purposes as the load at fracture divided by the original cross-sectional area of the specimen. All of the strength values reported in this paper are in terms of original area, that is in terms of engineering stress not true stress.

---

[1] Battelle Memorial Institute, Columbus, Ohio.

[2] Edgar C. Bain Laboratory for Fundamental Research, U. S. Steel Corp., Research Center, Monroeville, Pa.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON ELECTRON METALLOGRAPHY

New techniques and results of research studies in the field of electron metallography during the past year are featured in the papers presented in this Symposium which also includes the Sixth Progress Report of Subcommittee XI on Electron Metallography of Metals of Committee E-4 on Metallography which sponsored the Symposium.

These papers, including the Report, were presented at the Technical Session on Electron Metallography held on Tuesday, June 23, 1959, during the Sixty-second Annual Meeting of the Society at Atlantic City, N. J.

Mr. N. A. Nielsen, E. I. du Pont de Nemours and Co., Inc., acted as Symposium Chairman and Miss Anna M. Turkalo, General Electric Co., presided over the session.

The papers presented at this Technical Session are:

Introduction—N. A. Nielsen
Electron Microstructure of Precipitation-Hardenable Austenitic and Nickel-Base Alloys—Sixth Progress Report of Subcommittee XI on Electron Microstructure of Metals
Morphology of Phases in High-Temperature Alloys as Revealed by the Electron Microscope—J. R. Mihalisin
Simplified Electron Metallography of Steels—G. E. Pellissier
Techniques for Studying Structure and Growth of Tin Oxide by Electron Microscopy—P. S. Trozzo
Electron Metallography of Neutron-Irradiated Steels—R. F. McCartney and P. S. Trozzo
Electron Microscopy of Tin Plate—P. A. Stoll
Instrumental Modifications for Routine Electron Diffractometry—G. R. Grieger
Structure Analysis with Routine Electron Diffraction—G. R. Grieger
A Simple Application of the Carbon Replica Technique for Electron Metallography—L. Pellier
Electron Microscope Observations of Dislocations in Thin Metal Foils—R. M. Fisher and A. Szirmae
Effect of Heat Treatment on a Cast Nickel Alloy—C. G. Bieber and R. F. Decker
Composition of γ'^{2} Phase in High-Temperature Alloys from Electron Microprobe Measurements—L. S. Birks and R. E. Seebold

These papers and report, with the exception of the paper by L. S. Birks and R. E. Seebold, were issued as *ASTM Special Technical Publication No. 262*, entitled "Symposium on Electron Metallography."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON ELECTROLESS NICKEL PLATING

(Catalytic Deposition of Nickel-Phosphorus Alloys by Chemical Reduction in Aqueous Solution)

The need for publication of the information included in this symposium on Electroless Nickel Plating became evident a few years ago when a task group under Committee B-8 on Electrodeposited Metallic Coatings investigated the desirability of publishing a specification on electroless nickel plating. Section C, on Electroless Nickel, of Subcommittee I, was organized to develop the specification and a survey made by this group furnished information of such interest that it was decided to make it available for both technicians and scientists in the field; particularly for those not yet acquainted with this process and its problems.

This symposium is an attempt to gather all the known useful information about electroless nickel plating under one cover. It was also designed to be of particular value to those who are not yet acquainted with this process.

The papers included in the symposium are:

History of the Electroless Plating Process—Abner Brenner
Chemical Reactions—Gregoire Gutzeit
Characteristics of Deposits—W. H. Metzger, Jr.
Processing Procedures—Abraham Kreig
Advantages and Limitations—E. B. Saubestre
Applications—W. H. Safranke
Test Methods—Bennie Cohen
Patent Situation—Gregoire Gutzeit
Bibliography of References—C. F. Waite

These papers were issued as *ASTM Special Technical Publication No. 265* entitled "Symposium on Electroless Nickel Plating (Catalytic Deposition of Nickel-Phosphorus Alloys by Chemical Reduction in Aqueous Solution)."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON MICROSCOPY

This Symposium on Microscopy was sponsored by Committee E-1 on Methods of Testing. It covers most of the recent developments in light and electron microscopy and the effective application of this science to laboratory research and industrial production. It was held for the purpose of disseminating knowledge of progress and to emphasize the need for continuous refinements and improvements in microscopical techniques.

The papers in this symposium and their discussions were presented at the Twenty-ninth, Thirty-second, and Thirty-fourth sessions of the Sixty-second Annual Meeting of the Society, held on June 25 and 26, 1959, in Atlantic City, N. J.

Mr. F. F. Morehead, American Viscose Corp., Marcus Hook, Pa. acted as Symposium Chairman and presided over the first session. Messrs. M. C. Miller, Johns Manville Research Center, Manville, N. J., and L. L. Wyman, National Bureau of Standards, Washington, D. C., presided over the second and third sessions, respectively.

The papers included in the Symposium are:

Introduction—F. F. Morehead
Measurement with Phase and Interference Microscopes—Oscar W. Richards
Performance Characteristics of Objectives, Eyepieces, and Illuminators for Microscopy—H. W. Zieler
Chemical Microscopy in the Optical Industry—Germain Crossmon
The Study of Opaque Minerals in Reflected Light—Eugene N. Cameron
Application of Electron Microscopy in the Petroleum Industry—H. M. Allred
The Electron Microscope in the Study of Minerals and Ceramics—Joseph J. Comer
The Structure of Synthetic Fibers—Robert G. Scott
Microscopical Studies of Failure in Polymers—Sanford B. Newman
Microscopical Evaluation of Cotton Finishing Treatment—Mary L. Rollins, Ines V. deGruy, Verne W. Tripp, and Anna T. Moore

The papers presented at this symposium with the exception of the paper entitled "Interpretation of Light- and Electron-Microscopical Observations," presented by C. F. Tufts, Sylvania Electric Products, Inc., Bayside, L. I., N. Y., were issued as *ASTM Special Technical Publication No. 257*, entitled "Symposium on Microscopy."

AN APPROACH TO THE PROBLEM OF EVALUATION OF PLASTICITY AND WORKABILITY OF PASTES AND MORTARS*

By A. D. Conrow[1]

Synopsis

In this investigation an attempt was made to develop a means for evaluating some of the properties of pastes and mortars that influence their plasticity and workability. Measurements were made of the rates of deformation of hemispherical specimens of pastes and mortars when tested on the flow table. The deformation was recorded for each drop of the table and the change in per cent flow was used as a measure of the deformation.

It was found that the deformation was apparently related to the energy of the drop (weight of specimen times height of drop) by an equation of the type $F = AE^b$, where F = per cent flow, E = energy applied, and A and b are constants which appear to be related to ease of deformation and to cohesiveness (ability of the elements of the mass to cling together when the mass is deformed). The significance of the constants A and b, which are evaluated from the test results, is discussed.

Background Leading to Investigation

Over the past 35 years the author has seen many different types of tests proposed for evaluating the workability[2] of mortars and concretes. Since workability involves a number of different properties such as consistency, plasticity, cohesiveness, ease of deformation, and others, it seems hardly possible that a single number can evaluate workability of a paste, mortar, or concrete. In most of the proposed tests that were used to any extent, the specimen was made and then allowed to remain at rest for a period of time before being tested. It was observed that a mortar or concrete that appeared to have good workability as long as it was kept in motion would often lose it after a short period of rest. Some method of test was needed to evaluate workability while the material was in motion or agitation.

Deformation produced by a flow table simulates that in a concrete being puddled into place in a form. It was felt that the flow table action might be used to evaluate some of the factors involved in workability of pastes, mortars, and concretes. The flow table is in common use in many cement and concrete testing laboratories for evaluating consistency,

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Research Engineer, Ash Grove Lime & Portland Cement Co., Chanute, Kans.

[2] In the sense used in this paper, workability is conceived by the author to be that property of a mortar or paste which determines the ease with which it may be changed from one body shape to another body shape without appreciable change in the internal character of the body mass.

one factor connected with workability. In this investigation various pastes and mortars made with materials having observable differences in workability were tested on a standard 10-in. flow table (ASTM Specification C 230[3]). If differences in performance could be observed, could they be related to workability factors other than consistency? A method was needed for measuring the flow caused by each drop of the flow table without stopping operation of the table for measurement after each drop. Then a curve could be plotted relating flow to the energy assumed to be causing the flow.

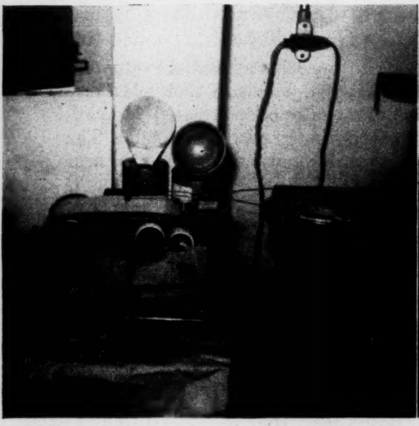


FIG. 1.—Ready for Start of a Flow Test.

FIG. 2.—Completion of a Flow Test.

FLOW MEASURING DEVICE

The flow for each drop was recorded with a scissors-like measuring device of very light-weight construction, having very light-weight shoes at one end which were moved by flow, or spread, of the specimen. The other end of the scissors was equipped with scribers such as are used with chart recorders. Before a test the shoes clasp the sides of the undeformed specimen and the scribers ride on a moving chart paper. During the test the shoes are forced apart by the spread of the specimen, and on the moving chart paper the scribers record the flow with each drop (see Figs. 1 to 3 and charts and data, Fig. 4).

TEST SPECIMENS

Tests were first made using the stand-

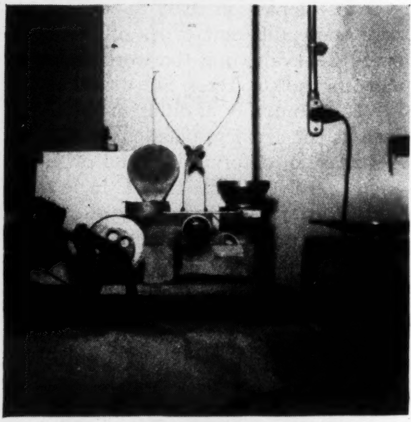


FIG. 3.—Equipment Used for Tests.

---

[3] Tentative Specification for Flow Table for Use in Tests of Hydraulic Cement (C 230 - 57 T), 1958 Book of ASTM Standards, Part 4, p. 196.

ard truncated-cone flow specimen. The flow-energy plots for this specimen were not easy to express in equation form, and the specimen shape was not simple for analyzing movement. A specimen in the form of a half-sphere 4 in. in diameter with base resting on the table surface was tested. A flow-energy plot for this specimen appeared to show a straight-line relationship for per cent flow, F, against energy units, E, when plotted on log-log coordinates within the range of ten drops of the table. The equation of such a curve has the form

$$F = AE^b \quad (1)$$

in which A and b are constants evaluated from the test results. If tests of different mortars yield equations with different values for A and b, the differences may be related to differences in the factors associated with workability. Paul S. Roller of the U. S. Bureau of Mines found such a relationship in his experiments on deformation of cylindrical mortar specimens.[4]

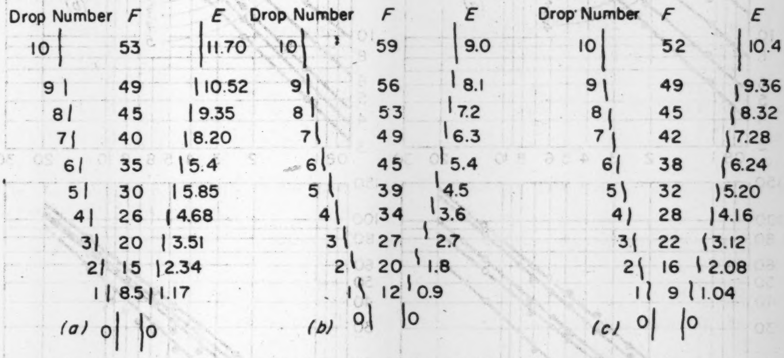


FIG. 4.—Traces of Flow Records for Mortars Indicated. Values for tabulation or curve plot entered on record.

(*a*) Type I portland cement
Curve 2, Fig. 5(*b*)
Standard flow 103 per cent
Weight of specimen = 0.585 kg
Measured flow at 10 drops = 56 per cent

(*b*) Masonry cement
Curve 2 Fig. 5(*e*)
Standard flow 105 per cent
Weight of specimen = 0.450 kg
Measured flow at 10 drops = 65 per cent

(*c*) Hydrated lime
Curve 2 Fig. 5(*f*)
Standard flow 100 per cent
Weight of specimen = 0.522 kg
Measured flow at 10 drops = 60 per cent

TEST PROCEDURE

The test procedures used to obtain flow records are given in Appendices I and II. Procedure A was used when values for standard flow were desired, and procedure B was used when values for standard flow were not desired.

RANGE OF MORTARS TESTED

Mortars containing the following water-powder paste mixtures were tested:

(1) A ground siliceous sand with a fineness of 96 per cent passing a No. 100 sieve. A mortar made with this material represents as poor a workability as is likely to be encountered for mortar-making materials (Fig. 5(*a*)).

---

[4] Paul S. Roller, "Plastic Flow of Dispersions and a New Approach to the Study of Plasticity," *Journal of Physical Chemistry*, Vol. 43, No. 4, April 1939.

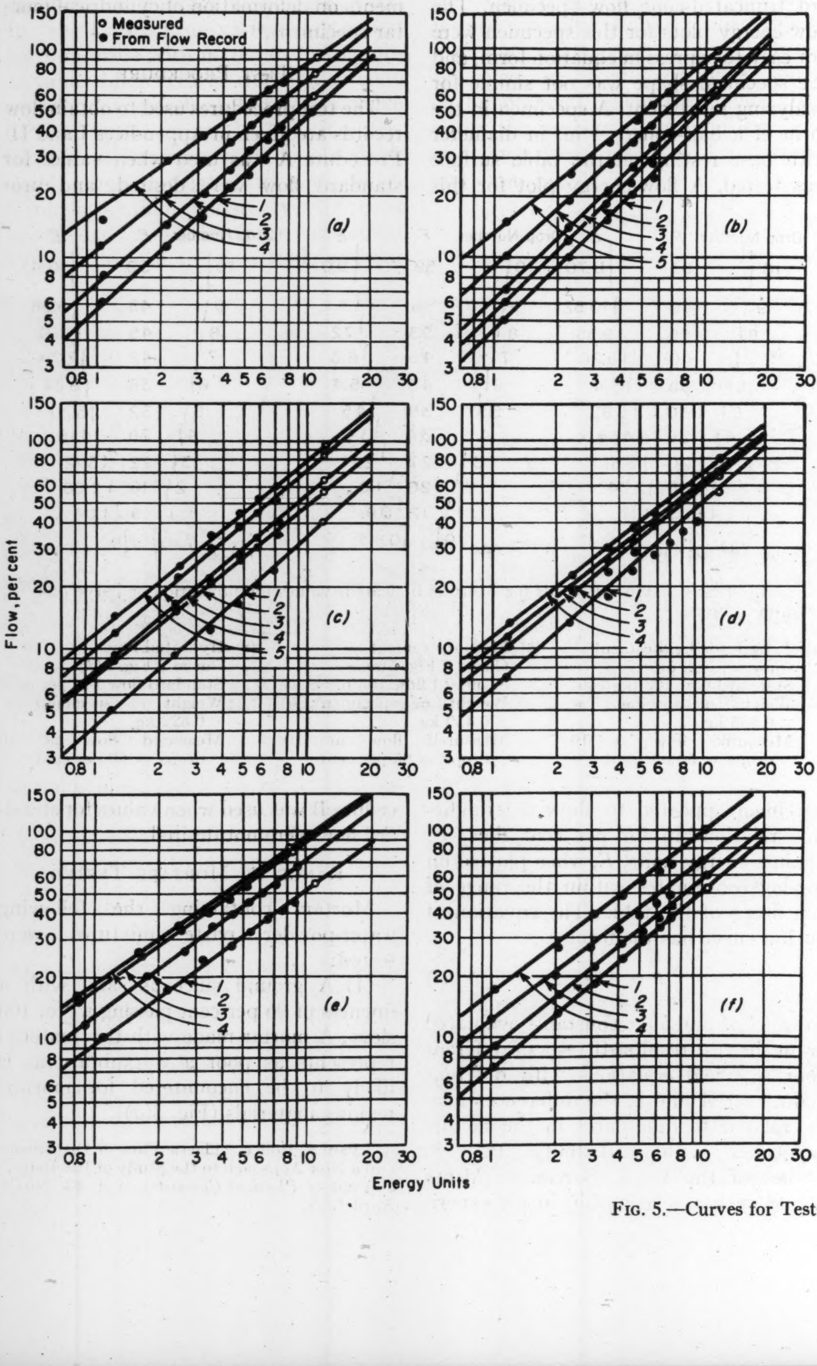


FIG. 5.—Curves for Tests

(*a*) Ground silica-sand mortar. ASTM Method C 109; Blaine meter specific surface approximately 2320.

Curve	Equation	Standard Flow, per cent	Specimen Weight, kg
1	$F = 5.5E^{0.93}$	80	0.525
2	$F = 7.3E^{0.89}$	102	0.535
3	$F = 10.8E^{0.83}$	130	0.535
4	$F = 16.5E^{0.70}$	145	0.530

(*b*) Coarse ground portland-cement mortar. ASTM Method C 109; Blaine meter specific surface approximately 2420.

Curve	Equation	Standard Flow, per cent	Specimen Weight, kg
1	$F = 4.4E^{1.0}$	95	0.570
2	$F = 5.3E^{1.0}$	110	0.570
3	$F = 6.1E^{0.98}$	125	0.578
4	$F = 9.0E^{0.94}$	140	0.583
5	$F = 13.0E^{0.83}$	150	0.575

(*c*) Normal type I portland-cement mortar. ASTM Method C 109; Blaine meter specific surface approximately 3200.

Curve	Equation	Standard Flow, per cent	Specimen Weight, kg
1	$F = 4.2E^{0.91}$	82	0.580
2	$F = 6.2E^{0.82}$	103	0.585
3	$F = 6.8E^{0.84}$	118	0.588
4	$F = 10.0E^{0.86}$	135	0.586
5	$F = 11.0E^{0.84}$	140	0.590

(*d*) Type III portland-cement mortar. ASTM Method C 109; Blaine meter specific surface approximately 4800.

Curve	Equation	Standard Flow, per cent	Specimen Weight, kg
1	$F = 6.0E^{0.91}$	100	0.578
2	$F = 8.7E^{0.82}$	110	0.584
3	$F = 9.7E^{0.80}$	116	0.586
4	$F = 11.2E^{0.79}$	127	0.595

(*e*) Highly plastic masonry-cement mortar. ASTM Specification C 91 (mortar for compressive strength); Blaine meter fineness approximately 6200.

Curve	Equation	Standard Flow, per cent	Specimen Weight, kg
1	$F = 9.4E^{0.73}$	94	0.505
2	$F = 12.8E^{0.73}$	105	0.450
3	$F = 17.0E^{0.72}$	113	0.430
4	$F = 18.0E^{0.70}$	118	0.420

(*f*) Lime-hydrate mortar. ASTM Methods C 110.

Curve	Equation	Standard Flow, per cent	Specimen Weight, kg
1	$F = 6.8E^{0.86}$	90	0.517
2	$F = 8.5E^{0.82}$	100	0.522
3	$F = 12.0E^{0.77}$	112	0.500
4	$F = 16.0E^{0.77}$	145	0.520

of Various Types of Mortars.

(2) A coarsely ground type I portland cement having a Blaine fineness meter specific surface of approximately 2420 sq cm per g. Such mortars have workability properties about the same as those obtained with the ground siliceous sand (Fig. 5(*b*)).

(3) Normal ground type I portland cement having a Blaine fineness meter specific surface of approximately 3200 sq cm per g. Such mortars have workability properties about the same as the average type I portland cement (Fig. 5(*c*)).

(4) Type III portland cement having a Blaine fineness meter specific surface of approximately 4800 sq cm per g (Fig. 5(*d*)). It is generally observable that workability properties of mortars made with type III portland cement appear to be better than those obtained with the average type I portland cement.

(5) A masonry cement having a Blaine fineness meter specific surface of approximately 6200 sq cm per g. Such mortars have very desirable workability properties, as needed in a mortar to be used for plaster or bedding masonry units (Fig. 5(*e*)).

(6) Hydrated lime. Hydrated-lime mortars have long been used for plaster and for bedding masonry units. Their properties are well known to persons familiar with plaster and masonry construction practices (Fig. 5(*f*)).

The compositions of the mortars containing the ground silica sand and portland cements were those specified in ASTM Method C 109.[5] For masonry-cement mortar the composition was as required in ASTM Specification C 91.[6] For lime mortar the composition was as required by ASTM Methods C 110.[7]

The above mortars were tested in accordance with procedure A of Appendix I. Other mortars were tested by the same procedure using various paste materials with river sand aggregate with gradation suitable for masonry mortar or plaster.

ASSEMBLY OF DATA

When tests are made in accordance with procedure A of Appendix I the following information is at hand for each flow-drop record taken:

1. Specimen composition,
2. Standard flow before taking flow-drop record,
3. Flow-drop record,
4. Weight of specimen,
5. Standard flow after flow-drop record, and
6. A caliper measurement of flow after 10 drops.

Data for Curve Plot:

To obtain the curves shown in Fig. 5, from which the values of A and b, Eq 1, can be obtained, values for per cent flow must be obtained from the flow record and the energy must be calculated for each drop. The values for per cent flow are obtained by measuring the width between parallel lines after each drop (Fig. 4) and subtracting the initial width at zero flow. The value obtained can be converted to per cent flow if the ratio of lever arms for shoes and scribers on the scissors measuring device is known. This device was constructed in such a way that the ratio of shoes lever to scriber lever was 40:25, therefore the per cent flow could be measured directly with the 40-divisions-per-in. scale on an engineer's scale. The lever-arm lengths were: shoe lever = 9.3 in. and scriber lever = 5.8 in.

---

[5] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 58), 1958 Book of ASTM Standards, Part 4, p. 124.

[6] Specifications for Masonry Cement (C 91 – 58), 1958 Book of ASTM Standards, Part 4, p. 29.

[7] Methods of Physical Testing of Quicklime and Hydrated Lime (C 110 – 58), 1958 Book of ASTM Standards, Part 4, p. 249.

The energy absorbed by the specimen in each drop is its kinetic energy just before table impact, which, neglecting friction losses, is equal to the change in potential energy during the drop, or, for one drop

$$E = Wh \qquad (2)$$

where:

E = energy of one drop, kg-cm,
W = weight of specimen, kg, and
h = drop distance, cm.

The drop distance was ½ in. (1.27 cm); therefore, for one drop

$$E = 1.27W \qquad (3)$$

and for N drops

$$E = 1.27WN \qquad (4)$$

This relationship neglects frictional losses in the table shaft and also neglects the change of energy of successive drops caused by a change of head of the mass as the specimen deforms. Actually the coefficient 1.27 can be assigned any value and the resulting calculated energy will be proportional to the energy causing deformation. A change in the value of the coefficient will not change the slope of the line through the plotted points. It will only shift the line in a direction parallel to the energy axis. It should be noted that a change in the coefficient will change the value of A in Eq 1. The value of 2 was selected for the coefficient to simplify calculations. Therefore, the energy values for plotting the curves in Fig. 5 were obtained by use of the equation

$$E = 2WN \qquad (5)$$

where:

W = weight of specimen in kg, and
N = number of drops.

It may be seen that the points obtained appear to fall on a straight line if allowance is made for experimental error. The value of A in Eq 1 can be read directly from the straight line, for it is the flow value at the point where the line crosses the unit-energy line. The value of b in Eq 1 is the slope of the line.

NOTE.—If tests are made by this method, energy calculations must be made using Eq 5 if the A values in Eq 1 are to be compared.

Significance of the Straight-Line Plot:

If the straight line relationship of flow against energy is accepted the significance of the constants A and b in the equation $F = AE^b$ is of interest.

The constant A is related to ease of deformation, or consistency. If we substitute the value 1 for E in Eq 1, then $F = A$, that is, A is the flow for application of the first unit of energy. Two mortars could be said to be deformable to the same degree by application of one unit of energy if the constant A in their equations is the same. They could also be said to have the same consistency if the constant A can be considered a measure of consistency.

The constant b in Eq 1 is related to the rate of deformation with continued application of energy; its value is determined by the slope of the line. A steep slope or high value of b indicates a rapid spread of the specimen, thus indicating a mortar that easily falls apart—that has poor cohesion and plasticity, both of which are factors affecting workability.

TEST RESULTS (*See* FIG. 5)

(*a*) Straight lines from tests of mortars made with materials like ground silica sand (*a*) and coarse ground portland cement (*b*) had relatively steep slopes—b values in the range of 0.90 to 1.00.

(*b*) Lines with slopes ranging from

TABLE I.—EQUATIONS OF MORTARS MADE WITH VARIOUS CEMENTING MATERIALS AND A SMOOTH-GRAINED RIVER SAND HAVING GRADATION DESIRABLE FOR PLASTERING AND MASON MORTARS.

Cementing Material	Mix, Lime-Cement-Sand	Equation	Standard Flow, per cent
Coarse ground portland paste	Paste	$F = 7E^{0.94}$	100
	Paste	$F = 10E^{0.85}$	130
Coarse ground portland mortar	0-1-3	$F = 6E^{0.91}$	95
	0-1-3	$F = 10E^{0.82}$	125
	0-1-3	$F = 15E^{0.80}$	150
	0-1-4	$F = 6E^{0.92}$	95
	0-1-4	$F = 8E^{0.89}$	115
	0-1-4	$F = 12E^{0.86}$	145
	0-1-5	$F = 6E^{0.91}$	95
	0-1-5	$F = 9E^{0.88}$	115
	0-1-5	$F = 11E^{0.86}$	140
Plastic masonry cement paste	Paste	$F = 10E^{0.76}$	95
	Paste	$F = 13E^{0.72}$	100
Plastic masonry cement mortar	0-1-3	$F = 10E^{0.72}$	94
	0-1-3	$F = 13E^{0.70}$	100
	0-1-3	$F = 17E^{0.62}$	109
	0-1-4	$F = 9E^{0.66}$	64
	0-1-4	$F = 16E^{0.62}$	106
	0-1-4	$F = 19E^{0.60}$	115
	0-1-6	$F = 11E^{0.56}$	60
	0-1-6	$F = 18E^{0.62}$	102
	0-1-6	$F = 20E^{0.60}$	110
	0-1-8	$F = 20E^{0.60}$	106
Hydrated lime paste	Paste	$F = 12E^{0.67}$	72
	Paste	$F = 18E^{0.67}$	105
Hydrated lime mortar	1-0-3	$F = 10E^{0.67}$	95
	1-0-3	$F = 13E^{0.67}$	100
	1-0-3	$F = 17E^{0.62}$	110
	1-0-5	$F = 12E^{0.67}$	92
	1-0-5	$F = 14E^{0.66}$	107
	1-0-5	$F = 18E^{0.64}$	125
	1-0-7	$F = 10E^{0.71}$	92
	1-0-7	$F = 12E^{0.70}$	105
	1-0-7	$F = 15E^{0.68}$	115
Lime-cement-sand mortar	1-1-6	$F = 15E^{0.74}$	124
	2-1-9	$F = 16E^{0.68}$	...
	1-2-9	$F = 16E^{0.72}$	120

0.80 to 0.90 were obtained for mortars made with the common type I portland cement (*c*). These mortars may be considered to be in a median range of plastic properties.

(*c*) Lines with slopes ranging from 0.60 to 0.75 were obtained for mortars made with a good masonry cement (*e*) or a plastic hydrated lime (*f*).

(*d*) The equations for mortars with

standard flow of 100 to 110 per cent containing pastes of materials like coarse ground portland cement differ considerably from the equations obtained for mortars with the same standard flow made with a good masonry cement or hydrated lime. There is a difference in both constants, A and b, even though mortars with the same standard flow are tested. The following equations illustrate differences observed from tests of a number of materials with standard flow in the range of 100 to 110 per cent.

Paste Material	Equation
Coarse ground portland cement	$F = 7E^{0.90}$
Type I portland cement	$F = 7E^{0.82}$
Good masonry cement	$F = 12E^{0.73}$
Hydrated lime	$F = 12E^{0.67}$

The above equations are from tests whose results are given in Table I.

(*e*) The value of b decreases somewhat for a mortar of a given dry composition as its consistency is changed by adding water. It may be that if a large proportion of the flow occurs with the first drop, later increments are smaller, giving a flatter slope to the line; or there could be a change in cohesive properties due to dilution of the paste. Either could reduce the rate of spread after the first drop and give a flatter slope to the line. Without a means of measuring cohesive properties it could not be determined whether one or both conditions contributed to the change in slope of the line.

(*f*) It was noted by direct observation that a portland-cement mortar having a standard flow of 100 per cent was more difficult to deform than a masonry-cement mortar with the same standard flow. It was also observed that two mortars whose equations had about the same constant A could be deformed with about equal effort. These observations are based on operator judgment because of lack of an accepted method for measuring ease of deformation. It is possible that this test may be suitable for this purpose.

(*g*) The masonry-cement mortar was air entraining in nature. The air content varied with variations in liquid content and with paste-aggregate ratio. This entrained air appeared to hold the change in the constant b to relatively low values as the paste content of the mortar decreased. When paste content was decreased the air content increased. The increased air appeared to compensate for decrease in paste, resulting in a lesser decrease in workability. This is believed to be the reason for only a small change in the constant b in Eq 1 for masonry-cement mortars as the paste content was varied.

(*h*) Specimens of hydrated-lime mortars contained little air and were heavier than masonry-cement mortar specimens. Variations in weight with changes in liquid content were less. The value of b increased appreciably as paste content was decreased.

DISCUSSION

Workability characteristics of the mortars tested differ through a range as great as is likely to be encountered in the common use of such materials. The mortars were selected with the idea that they would be familiar to practically all who are interested in the use of such materials in construction.

In Fig. 5, it may be seen that the points drift off the curve as the number of drops increases. This drift is a result of the fact that the shoes of the measuring device are not pushed out to the full flow value, and this inaccuracy increases as the cake of mortar becomes thinner. The point obtained by measurement with calipers at the tenth drop (open circle) shows where the trend of points should have been, had the device measured the flow accurately. Therefore, the curve is best defined by a line through the points for the first five drops and through the open circle. Curve 4 of Fig. 5(*a*) shows the point for the first drop considerably off the curve. In this case the mortar slumped to a flow of about 5 per cent from its own weight, indicating a mortar beyond the range in which this test can be considered applicable.

The indication that there is a straight-line log-log relationship between per cent flow and applied energy provides a means for recording the relationship in equation form. The following information appears to be given by that equation:

(*a*) The constant A in the equation indicates consistency characteristics with ranges as follows: (1) If A ranges from 5 to 8 consistency will be similar to that of a common type I portland cement mortar with 100 to 110 per cent flow (Method C 109). (2) If A is in the range of 12 to 16 the consistency of the mortar will be similar to that of a good masonry-cement mortar with 100 to 110 per cent flow (Specification C 91) or a hydrated-lime mortar with 100 to 110 per cent flow (Methods C-110).

(*b*) The constant b in the equation appears to indicate plastic and cohesive characteristics with ranges as follows: (1) If b is in the range of 0.90 to 1.0 the plastic and cohesive character will be similar to that of a mortar made with a coarse ground portland cement having a fineness represented by Blaine fineness meter specific surface of 2200 to 2500 sq cm per g. (2) If b is in the range of 0.80 to 0.90 the plastic and cohesive character will be similar to that of a mortar made with portland cement ground to a fineness represented by a Blaine fineness meter specific surface of 3000 to 3300 sq cm per g. (3) If b is in the range of 0.70 to 0.80 its plastic and cohesive character will be similar to a mortar made with the common, fine-ground, high-early-strength portland cement. (4) If b is in the range of 0.60 to 0.70 its plastic and cohesive character will be similar to mortars made with good masonry-cement, lime-cement combinations, and plastic hydrated-lime mortars.

TESTS TO SHOW EQUATION CHANGES FROM VARIATIONS IN CHARACTER OF MORTARS AND PASTES

Table I shows changes in the constants in Eq 1 resulting from the use of different kinds and quantities of mortar constituents. All mortars contained aggregate from the same lot. The aggregate was a well-graded, smooth-grained river sand having a gradation desirable for plaster or masonry mortar. Mortar mixes for some paste-making materials were varied through proportions ranging from rich to lean and consistencies stiff to soft, to learn whether changes in paste content would cause changes in b. A change in b can be seen in mortars containing hydrated lime with little air entrainment. The masonry cement tested was one with high sand-carrying capacity. Thus there should be little change in b through the range of mixtures tested if b evaluates plastic quality. For mortars made with this cement the decrease in paste content was compensated by an increase in air content.

The data in Table I show that masonry and plaster mortars will be in the consistency range desired by the workman when A has values ranging from 12 to 18. If the equation for a mortar shows A to be outside that range the consistency will probably not be suitable for use.

INVESTIGATIONS IN WHICH THIS TEST MAY BE USEFUL

(1) Change in consistency with any variation of conditions expected to change consistency.

(2) Stiffening rate with prolonged mixing or time after addition of mixing water.

(3) Sand-carrying capacity. If b exceeds 0.8 it is probable that sand-carrying capacity has been exceeded.

(4) Change in cohesiveness with additions of admixtures and surface active agents, change in fineness of grinding, prolonged or intensive mixing, and day-to-day or hour-to-hour runs during manufacture.

COMMENTS

This test can be made as quickly as a flow test once the equipment is set up for the test.

The equation of the curve may be found quickly if recording scissors are constructed in such a way that flow values can be read directly from an engineer's scale and points are plotted on log-log coordinates having a scale such that one log cycle is 100 divisions for some scale of an engineer's scale.

Repeat tests can be made quickly to check what appears to be abnormalities. During testing for this investigation reproducibility was very good where stiffening rate was slow.

A record of the test is obtained on the chart paper, which may be analyzed at leisure, filed away for record, and reexamined if values are questioned later.

SUMMARY AND CONCLUSIONS

1. The standard flow table fitted with auxiliary measuring and recording equipment provides a fairly simple means for measuring the rate and extent of deformation of paste or mortar specimens of suitable shape.

2. The deformation (per cent flow) produced under the conditions of this test appears to be related to the energy causing deformation (generated by fall of the specimen) by an equation of the form $F = AE^b$ in which F is per cent flow, E is an energy factor proportional to the energy of fall of the specimen and A and b are constants related to factors affecting workability of the specimen material. Analysis of the test results indicates that A is closely related to the consistency of the material under test, and b appears to be related to other factors affecting workability such as cohesion and resistance to continued deformation.

3. The test develops two values for a given paste or mortar. One is related to the ease with which the mass may be deformed. This is considered to be a measure of consistency. The other is related to a property which may be called cohesion—a property considered to be necessary if a mortar is to have good plasticity.

4. The method of test and interpretation described may be found useful in a variety of investigations involving changes in properties associated with workability. The changes may be influenced by fineness of the powder from which the paste is made, liquid content, mix proportions, admixtures, length of mixing time, and other factors.

APPENDIX I

TESTING PROCEDURE A—WHEN STANDARD FLOW DATA ARE DESIRED

1. Prepare mortar or paste. Batch size of 700 g of dry material is sufficient.

2. Mix by machine in accordance with Method C 305.[8] Use enough water to give a standard flow of 95 to 100 per cent.

3. Mold in a standard flow mold and obtain standard flow in accordance with Specification C 109.[5] Record flow.

4. Return mortar to mixing bowl. Remix for 15 sec at intermediate speed. Fill half-sphere mold, weigh specimen and tared mold. Then obtain flow-drop record for 10 drops. Measure flow at 10 drops with calipers and record.

---

[8] Tentative Method for Mechanical Mixing of Hydraulic Cement Mortars of Plastic Consistency (C 305 – 58 T), 1958 Book of ASTM Standards, Part 4, p. 171.

5. Return mortar to mixing bowl, remix for 15 sec at slow speed. Obtain standard flow and record.

6. Return mortar to mixing bowl. Add enough water to give standard flow of 100 to 110 per cent. Remix 15 sec at medium speed, then obtain standard flow.

7. Continue procedure indicated above (steps 4 through 6), adding water in stages to increase flow in increments of about 10 per cent until mortar acquires a consistency close to that at which it will slump under its own weight.

By the above procedure 3 to 5 flow-drop records on the mortar under test may be obtained from which plots of per cent flow against energy may be made.

APPENDIX II

TESTING PROCEDURE B—WHEN STANDARD FLOW DATA ARE NOT DESIRED

1. Prepare mortar or paste. Batch size of 700 g of dry materials is sufficient.

2. Mix by machine in accordance with Specification C 305.[8] Use enough water to give consistency appearing suitable for plaster or masonry use or a consistency for which test data are desired.

3. Fill half-sphere mold. Weigh specimen and mold. Place specimen on flow table and obtain flow-drop record for 10 drops. Measure flow at 10 drops with calipers and record.

4. If data on a repeat test without further addition of water are desired, return mortar to mixing bowl. Remix 15 sec at slow speed.

5. Repeat step 3.

6. Further tests may be made as desired with or without adding increments of water. If increments of water are added the 15-sec remix should be at medium speed.

DISCUSSION

MR. HUBERT WOODS[1] (*presented in written form*).—This paper offers a refreshingly different and useful approach to workability. The author properly abandons the outworn view that the subjective concept we call "workability" is measurable by a single number. He recognizes that "workability" is a complex concept involving at least two and perhaps four or five underlying properties, some of which are themselves not readily definable in terms of the basic units mass, length, and time.

The equipment and procedures used by the author are disarmingly simple, but it is obvious that many months of careful study and trial must have gone into their development. The results appear to justify the conclusion that he has arrived at a useful means for studying some of the factors related to workability.

It is noted that procedure A of Appendix I was used to obtain the results shown in the paper. However, an alternative procedure B is described in Appendix II. Presumably, procedure B will produce test mortars having about the same values for the constant A in Eq 1 whereas procedure A will not.

I hope that the author will discuss the practical differences in results of these alternate methods, and will also discuss the apparent challenge to the validity of the usual method of determining consistency with the flow table.

MR. A. D. CONROW (*author*).—Mr. Woods asks if there will be any difference in the value of A in the equation $F = AE^b$ if procedure A is followed in one case and procedure B in another case.

---

[1] Director of Research, Portland Cement Assn., Research and Development Laboratories, Skokie, Ill.

Since the value of A in the equation $F = AE^b$ is a measure of consistency, the only difference in this value obtained from the two procedures would be any change in consistency caused by the extra manipulation required to obtain the standard flow when procedure A is employed. The extra manipulation for procedure A causes very little change in consistency, thus, there is very little difference in the A value obtained by the two methods.

The reason for setting up procedure B, which differs from procedure A only in the omission of determination of standard flow, is that it would be used after the user of this test becomes familiar with the consistency represented by the various A values obtained. When this familiarity has been acquired the standard flow is no longer needed.

Procedure A was set up for this paper to show the relationship of the standard flow to the A value in the equation to persons familiar with the consistency represented by the standard flow.

Mr. Woods further asks that I discuss the apparent challenge to the validity of the usual method for determining consistency with the flow table. The author has observed many times that the mortar made with the common type I portland cement which shows a consistency indicated by 100 per cent flow under conditions of the standard flow test which employs twenty-five $\frac{1}{2}$-drops will have a stiffer consistency, that is, is less easily deformed, than a masonry cement mortar or a lime mortar which shows 100 per cent flow under conditions of the standard flow test.

A reason for this may be given as follows: the mortar specimen made from the type I cement will be considerably heavier than the specimens made from masonry cement or a lime mortar. Thus 25 drops will result in the expenditure of considerably more energy to cause deformation when the heavier specimen is tested, and accordingly a stiffer mortar would be caused to show the same flow as a less stiff mortar in a specimen having less weight.

The A values in the equations obtained by the testing procedure which is the subject matter of this paper will show better relative consistency relations because they represent deformations caused by practically the same energy expenditure.

MR. F. J. MARDULIER.[2]—The Working Committee on Bleeding, Plasticity, and Workability of ASTM Committee C-1 on Cement has for some time been working on the problem of measuring that elusive quality of cement mortars and concrete called workability. Workability we have come to agree consists of various component parts. The thing that interests me particularly about the author's paper is that I believe he has measured possibly the most elusive component of those various components that go to make up workability. He has I believe measured the plasticity component and, furthermore, has a built-in consistency meter as well, namely, the constant A in the equation:

$$F = AE^b$$

I think the author is to be commended for a substantial contribution to the study of workability. We have seen many tests, we have been considering many tests; we have seen none that measures more than one factor and clearly states what these factors are. As one can see from the author's equation, the two factors are there plainly visible. Although not everyone may agree that these two factors are "consistency" and "plasticity," still I believe they must be considered significant factors in the measurement of workability.

---

[2] Manager, Rock Products Chemicals Dept., Dewey & Almy Chemical Co., W. R Grace & Co., Cambridge, Mass.

The author has mentioned the simplicity of adapting this test to use in any cement laboratory, another decided advantage of the test. At this point, I should like to ask the author one question.

When you use a stiff mortar such as the one giving a consistency or A factor of 0.82, what would happen to the linearity of the curve, if you had jigged the mortar more than 10 jigs on the flow table.

MR. CONROW.—I was unable to get a very accurate measurement where the flow flows out to a thin cake. So I have considered that this test is only applicable for the change in form which takes place under the 10 drops. Now if a sufficiently accurate measure of change in flow can be devised, extension of measurement beyond 10 drops may become feasible $\frac{1}{m}$ I have thought that electronic devices or something like that might be devised to make such measurements but would probably be too expensive for this operation. I do not know whether such measurements would continue to show a straight line relationship.

MR. MARDULIER.—I suggest for the consideration of ASTM Committee C-1 that there may be an indication here of the desirability of limiting the total number of jigs on the flow table to 10 rather than the present 25. I think it worthwhile considering.

MR. CONROW.—A mortar showing an A value of 0.82 in the equation $F = AE^b$ would not be plastic, and thus would be outside the range where this test method would be applicable. An A value of 1 or less indicates a mortar which shows a flow of 1 per cent or less for the first drop of the table. A mortar this stiff would probably crumble. For this reason no tests were made on mortars as stiff as would be represented by an A value of 1 or less.

It is possible the 0.82 value given has been confused with the values for the exponent b in the equation.

The test procedure was used on mortars through the range too stiff to be usable to mortars too soft to be usable. The A values through this range were in the range 4 to 20. A stiff mortar would, of course, show a low flow at 10 drops, and when drops were continued as far as sufficiently accurate measurements could be obtained the flow-energy relationship still appeared to be linear on log-log coordinates.

For most mortars in the consistency range considered usable, the mortar cake on the table had become so thin after 10 drops that the measuring device did not give a sufficiently accurate measure of the flow. Furthermore, the plotted points on log-log paper after 10 drops become so close together that they add little to the determination of the slope of the curve.

MR. HANS F. WINTERKORN.[3]—I found this paper and also the remarks of Mr. Mardulier very intriguing. In order to follow up these ideas which Mr. Mardulier and others undoubtedly found in this presentation, it would be important not only to know the energies applied to the system but also the energy used by the system, and that is the more difficult problem.

I would not have said anything here at all if Mr. Mardulier had not brought out the possible extension. I saw it also and I think it very interesting, but I do not think it is as easy as it appears because the amount of energy used is a little bit more difficult to determine.

MR. C. C. CONNOR.[4]—There is strong

---

[3] **Professor of Civil Engineering, Princeton University, Princeton, N. J.**

[4] **Consultant, Masonry Construction and Maintenance, Verona, N. J.**

evidence that the grading of the sand affects the workability of mortar in the field. I would like to ask the author if he has found any indications that the grading of the sand in the mortar influences the flow table results.

MR. CONROW.—There are still a number of fields which may be explored by use of this test. I have not explored the effect of different sand gradations. The range involved in these tests is that which differs by the amount that the grading of graded Ottawa sand differs from a good masonry sand used in our area. Nothing significant could be observed in this range of difference. I have thought that exploration of effect of sand grading using this method might give interesting information.

MR. E. J. WECHTER.[5] In Table I it is noted that the values for *b* are sometimes higher in the leaner mixes (1:4 and 1:5 mixes). I wonder if you would comment on this.

MR. CONROW.—There was some work done to explore the effect of decreasing paste content on the terms in the equation. Some results are shown in Table I. When non air-entraining mortars are under test and comparisons are made of mortars of the same consistency there are indications that the slope of the flow-energy curve increases as the amount of paste-making powder in the mix decreases. This results in an increase in the *b* term of the equation. This is thought to indicate a less desirable degree of workability.

When air-entraining mortars were under test it was found that as the paste-making powder in the mix was decreased the air entrainment increased. The air increase compensated for powder decrease, and there was little loss in workability and only insignificant change in curve slope or the *b* term in the equation.

It may be possible that this test could be used to measure the sand-carrying capacity of a selected paste-making material. It could be said that this limit was reached when the slope of the curve, that is, the *b* term, exceeds 0.75.

MR. MAX E. GRUNEWALD.[6]—I would like to ask the author how sensitive this test is for the relatively minor variations which would be expressed on the water retention scale of say 80 to 85 per cent.

MR. CONROW.—It is believed you have in mind the question, "Will this test have a value for keeping check on hour-to-hour and day-to-day production of a product?"

I have not carried through check runs of this nature since most of the work connected with this report has been development of the test method. The development work indicates the test might have merit for keeping check on production, for if there was appreciable change in curve slope in mortars for which the *A* term of the equation was practically the same the change might indicate a change in the nature of the product.

The range of slope from very plastic and workable to nonplastic and very poorly workable was 0.5 to 1.0. From the curve plots it is easy to read changes in slope of 0.01. It could be said this would give a range of 50 points from very good to quite bad. A change of 5 points could be considered significant as illustrated by a change from 0.65 to 0.70 in slope.

Since water retention is not a direct measure of workability, I have not established a direct relationship of the results of this test to water retention.

(*Author's closure*).—I appreciate the interest shown in the development of this test procedure and the information it may give in the field of evaluation of workability of pastes and mortars. This interest is indicated by the various discussions and questions submitted.

---

[5] Division of Research, Louisville Cement Co., Speed, Ind.

[6] President, Coplay Cement Manufacturing Co., Coplay, Pa.

The paper deals largely with the development of the test procedure and the information gained during development. The indicated information given during development suggests use of the test method to explore other fields in which its use may give desired information such as hour-to-hour or day-to-day check for quality control during production, effect of variations of aggregate grading, effect of use of surface active agents, and by extension to larger equipment to tests of concrete on the 30-in. flow table.

Considerable thought has been given to the point made by Mr. Winterkorn and it is recognized that it may be very difficult to analyze the action of the forces and energy causing deformation under the conditions of this test. The accuracy of measurement obtained with this test is not good enough to be positive that the flow-energy curve if accurately obtained would show the indicated linear relationship of flow to energy. It is very interesting to find that with the accuracy obtainable by the methods in this test there appears to be a linear flow-energy relationship when plotted on log-log coordinates which indicates $\log F = \log A + b \log E$ and the terms A and b appear to have significance with respect to properties related to workability of pastes and mortars.

QUANTITATIVE DETERMINATION OF THE FOUR MAJOR PHASES IN PORTLAND CEMENTS BY X-RAY ANALYSIS*

By Stephen Brunauer,[1] L. E. Copeland,[1] D. L. Kantro,[1]
C. H. Weise,[1] and Edith G. Schulz[1]

Synopsis

A method has been developed for the direct determination of the four major phases (tricalcium silicate, dicalcium silicate, tricalcium aluminate and calcium aluminoferrite) as well as of the composition of the calcium aluminoferrite phase in portland cements by X-ray quantitative analysis.

This method, which employs no assumptions about the compositions of the phases, has been compared with the previously reported combined X-ray and chemical analysis method, which does employ certain assumptions about compositions. It has been found that the results for three of the four phases are in good agreement and for one (tricalcium silicate) in fair agreement. The X-ray method results are systematically higher than the combined method results for all phases except tricalcium aluminate, for which the methods are in agreement.

A comparison of the sums of the phases with the sums of the oxides obtained from chemical data shows that the combined method gives somewhat more accurate results than the X-ray method. Using the combined method values for tricalcium and dicalcium silicate, new calibration constants have been obtained, and the X-ray method values have been recalculated for the silicates with the new constants.

Because the combined X-ray and chemical analysis method is the most accurate method available at present for the estimation of the four phases, it has been adopted as a standard, and the values for the phases obtained by the three other available methods (X-ray method, microscopical analysis and potential compound calculation) have been compared with it. It is concluded that the X-ray method gives more accurate results than the two older methods. In view of this, the X-ray method appears to have possibilities to replace chemical analysis and potential compound calculation in industrial practice.

Recently, a method was presented for the quantitative determination of the four major phases in portland cements (**1**).[2] The phases are tricalcium silicate or alite, beta-dicalcium silicate or belite, tricalcium aluminate and calcium aluminoferrite. Alite and belite, as used here, are tricalcium silicate and beta-dicalcium silicate containing small amounts of alumina and magnesia. The method was based on a combination of data obtained from chemical analysis and X-ray quantitative analysis; it will be referred to as the combined method. Prior to the development of the method, two others were available for the estimation of the four major phases: the method of Bogue (2), with its numerous modifications, and the method of microscopical analysis (3). The first of these is an indirect method: the composition is not measured, but is calculated on the basis of the amounts of calcium oxide, silicon dioxide, aluminum oxide and ferric oxide obtained by chemical analysis. It does not give the actual quantities of the phases, but the potential quantities that would be present if certain conditions existed in the kiln and during cooling of the clinker. The second is a direct method, but it is not very accurate, because the smallest particles are either invisible or, at least, difficultly identifiable in the optical microscope.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Manager, Senior Research Chemist, Research Chemist, Assistant Research Chemist, and Assistant Research Chemist, respectively, Basic Research Section, Portland Cement Assn., Research and Development Laboratories, Skokie, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Bogue's method of potential compound calculation involves four assumptions: (1) all the ferric oxide is in the ferrite phase; (2) the ferrite phase is tetracalcium aluminoferrite (or brownmillerite); (3) all the aluminum oxide not in the ferrite phase is in tricalcium aluminate; and (4) all combined silica is in the two calcium silicates. The combined method retains assumptions (1) and (4), but discards assumptions (2) and (3). It gives the quantities of the four major phases more accurately than the older methods; at the same time, it also gives the composition of the ferrite phase (1).

The present paper reports a method for the quantitative determination of the four phases by X-ray analysis alone, referred to as the X-ray method. Because the combined method involves certain assumptions as to the compositions of the phases, it is, in part, an indirect method. The X-ray method is entirely direct; it employs no data of chemical analysis and no assumptions as to the compositions of the phases. The one assumption involved in the method is that the calibration data obtained for the pure phases are applicable for the impure phases found in portland cements. In other words, the assumption is that the slight amounts of impurities probably present in all four phases do not alter appreciably the intensities of the X-ray diffraction lines used in the analysis.

The X-ray method has practical possibilities; it may eventually be substituted for the chemical analysis and potential compound calculation now used by the portland-cement industry. The X-ray method is more accurate, and, probably, with some practice it can be made less time consuming.

EXPERIMENTAL PROCEDURES

As was described in detail in a previous paper (1), the tricalcium aluminate and calcium aluminoferrite calibrations were carried out with mixtures of alite, belite, tricalcium aluminate, and one of a group of four laboratory preparations of different compositions in the ferrite solid solution range. These compositions were $2CaO \cdot Fe_2O_3$, $6CaO \cdot Al_2O_3 \cdot 2Fe_2O_3$, $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$, and $6CaO \cdot 2Al_2O_3 \cdot Fe_2O_3$.

The alite-belite calibration was carried out with laboratory preparations of mixtures having alite-belite ratios varying from 25:1 to 1:6, as well as with almost pure alite and almost pure belite. These materials were prepared by adding small amounts of Al_2O_3 and MgO to the raw mix in order to stabilize the calcium silicate structures in the modifications found in portland cements.

The same 20 cements investigated by the combined method were reevaluated by the present method. These consisted of 12 so-called "long time study" (LTS) cements, 4 "special basic research"

(SBR) cements, 2 special Belgian (B) clinkers, furnished by W. L. De Keyser and A. van Bemst of the University of Brussels, and 2 Lone Star Cement Co. (LS) cements furnished by M. A. Swayze of that company. These last two cements were specially designed to contain no tricalcium aluminate. Detailed descriptions and analyses of the LTS and SBR cements have been published elsewhere (3–7).

The data were derived from the same X-ray diffraction charts used for the determination of phase compositions by the combined method. The charts themselves were obtained with a North American Philips Geiger counter diffractometer. The radiation used was CuK_α.

The method of X-ray specimen preparation was that described by Copeland and Bragg (8), with certain modifications. Samples were placed in brass holders through a small metal sieve, both of which were electrically grounded to a water pipe. The sample was kept in the sieve for 10 min while the sieve stood on a grounded copper plate. The sample was next sifted into the grounded specimen holder which incorporated a brass rather than glass cover plate. The purpose of this grounding procedure was to permit static electrical charge to leak off the particles of the sample. It was found that unless these precautions were taken, sometimes spurious results were obtained. The effect of electrical charge on particles of cement and of the silicon internal standard may be to cause separation of the mixture, at least in the surface layers, where homogeneity is most important.

The manner in which the data were obtained from the diffraction charts was the same as in the combined method. The same diffraction lines were used: at about 2.65 Å for ferrite, at 2.71 Å for tricalcium aluminate, and at 2.20 and 1.77 Å for alite and belite. These last two are composite lines consisting of both alite and belite contributions. For the relative intensity determinations, the 3.138 Å diffraction line of silicon was used as the internal standard. For ferrite *d*-spacing determinations, the same line and the 1.920 Å silicon line were used. Some of the *d*-spacing determinations were also carried out using the 3.300 Å and 2.333 Å lines of potassium bromide as references.

CALIBRATION AND DETERMINATION OF CALCIUM ALUMINOFERRITE AND TRICALCIUM ALUMINATE

The calibrations of calcium aluminoferrite and tricalcium aluminate were described in detail in the earlier paper (1). The calcium aluminoferrite calibration led to the equation

$$P_F = P_O[(\sigma_0 + \sigma_1\nu + \sigma_2\nu^2)R_F + (\varphi_0 + \varphi_1\nu + \varphi_2\nu^2)R_A] \ldots\ldots (1)$$

where P_F and P_O are the weight fractions (or percentages) of ferrite and internal standard, respectively; R_F and R_A are the intensity ratios of ferrite and tricalcium aluminate to internal standard, respectively; ν is the molar Al_2O_3/Fe_2O_3 ratio in the ferrite phase, and σ_0, σ_1, σ_2, φ_0, φ_1, and φ_2 are constants having the values 2.920, −2.111, 0.978, 0.152, 0.024 and 0.072, respectively.

Equation 1 contains two unknowns, P_F and ν, which determine the amount and the composition of the ferrite phase. In one version of the combined method, the equation was solved simultaneously with an equation relating P_F and ν with the ferric oxide content of the cement. Thus, the X-ray data embodied in Eq 1 were combined with chemical data to obtain P_F and ν.

In the second version of the combined method, different X-ray information was used. The value of ν can be obtained not only from X-ray diffraction line intensity measurements but also from the position (d-spacing) of the peak of the ferrite line. This value of ν, called ν_d, combined with the chemical information described above, gave another value for P_F. The two values of P_F and the two values of ν were then averaged.

In the X-ray method, the value of ν_d is used in Eq 1 to obtain P_F. Thus, the composition and the amount of ferrite are obtained without the use of any chemical data.

TABLE I.—FERRITE COMPOSITIONS AND FERRITE AND TRICALCIUM ALUMINATE CONTENTS BY VARIOUS METHODS.

Cement	ν, Combined	ν, X-ray	P_F, Combined, per cent	P_F, X-ray, per cent	P_F, Microscopical, per cent	P_F, Potential, per cent	P_A, Combined, per cent	P_A, X-ray, per cent	P_A, Microscopical, per cent	P_A, Potential, per cent
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*
LTS-11.....	1.05	0.73	7.4	9.2	8.4	7.3	8.4	7.6	5.3	10.8
LTS-12.....	1.01	1.00	7.3	7.4	5.2	7.3	7.6	7.6	8.5	11.3
LTS-15.....	0.98	1.15	7.5	6.5	5.1	7.6	9.9	10.3	9.5	10.1
LTS-17.....	0.85	0.76	8.8	9.6	6.0	9.4	7.6	7.2	6.8	9.1
LTS-18.....	1.24	1.44	7.7	7.2	5.4	7.0	7.4	7.6	6.1	12.3
LTS-23.....	0.69	0.74	14.2	13.3	8.0	16.4	2.3	2.7	2.6	2.3
LTS-25.....	0.83	0.80	13.8	14.1	14.3	14.9	0.2	0.0	1.7	3.9
LTS-31.....	1.08	1.17	6.6	6.2	4.8	6.4	7.4	7.6	7.3	9.7
LTS-33.....	1.09	0.92	7.9	8.7	4.1	7.6	7.4	7.0	9.8	9.6
LTS-41.....	0.90	0.86	14.6	15.0	18.3	15.2	0.2	0.0	1.9	3.7
LTS-42.....	1.16	1.16	8.8	10.0	5.4	8.2	1.3	1.3	0.9	2.9
LTS-51.....	0.84	0.76	9.3	10.1	9.2	10.0	0.6	0.2	1.0	2.6
SBR-15754..	1.44	1.44	8.0	9.7	...	6.7	6.9	6.9	...	12.4
SBR-15622..	0.81	0.71	11.7	13.0	...	12.8	0.0	0.0	...	3.2
SBR-15497..	0.86	0.90	7.4	7.2	...	7.9	8.8	8.9	...	9.4
SBR-15669..	1.11	1.22	6.1	5.8	...	5.8	0.8	1.0	...	1.9
LS-1........	0.38	0.30	11.4	13.1	...	...	0.0	0.0	...	...
LS-2........	0.37	0.30	10.8	12.2	...	...	0.0	0.0	...	...
B-H........	0.75	0.83	13.2	11.8	...	14.9	6.4	6.9	...	7.1
B-L........	0.94	1.07	8.6	7.7	...	8.8	8.9	9.3	...	9.4

The earlier paper (1) gave comparisons between the results of the combined method and those of the two older methods. In Table I, the results of the X-ray method are compared with those of the combined method. Column 3 shows that, disregarding the two LS cements which were especially designed to have low alumina contents, the range of X-ray ν values is from 0.71 to 1.44. This is almost the same as the range of the combined method ν values in column 2, 0.69 to 1.44.

The median X-ray ν value is 0.92, as compared to the median combined method value of 0.96. Thus, both methods indicate that, on the average, the ferrite phase in portland cement has a composition close to $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$.

In Table I, columns 4 and 5, the combined and X-ray values for ferrite content are given. A comparison shows that the X-ray values are, on the average, slightly higher, the systematic difference being 0.3 per cent. The average discrepancy between the two methods is 0.9 per cent.

The calibration equation for tricalcium aluminate is

$$P_A = P_O\mu[R_A - (\tau_0 + \tau_1\nu + \tau_2\nu^2)R_F] \ldots (2)$$

where P_A and P_O are the weight fractions (or percentages) of tricalcium aluminate and internal standard in the cement, respectively, R_A and R_F are the intensity ratios of tricalcium aluminate and calcium aluminoferrite to internal standard, respectively; ν is the molar Al_2O_3/Fe_2O_3 ratio in the ferrite phase; and μ, τ_0, τ_1 and τ_2 are constants, having the values 1.459, 1.143, -1.327 and 0.444, respectively.

In the combined method, Eq 2 was solved for P_A using each of the two values of ν obtained in the ferrite calculations. The value of P_A was taken as the average of the two results. In the X-ray method, only the ν_d values are used in Eq 2.

Columns 8 and 9 of Table I show the tricalcium aluminate contents of the 20 portland cements, obtained by the X-ray and the combined methods. There is no systematic difference between the two methods, and the average discrepancy is only 0.2 per cent.

TABLE II.—ALITE AND BELITE CONTENTS BY VARIOUS METHODS.

Cement	P_α, Combined, per cent	P_α^0, X-ray, per cent	P_α, X-ray, per cent	P_α, Microscopical, per cent	P_α, Potential, per cent	P_β, Combined, per cent	P_β^0, X-ray, per cent	P_β, X-ray, per cent	P_β, Microscopical, per cent	P_β, Potential, per cent
1	2	3	4	5	6	7	8	9	10	11
LTS-11	57.3	62.2	60.0	56.1	54.8	19.0	20.7	19.9	12.4	18.0
LTS-12	51.0	49.2	48.4	47.3	48.9	27.7	26.8	26.1	22.2	24.5
LTS-15	61.0	66.8	63.4	56.2	72.4	12.3	13.4	13.0	6.0	3.3
LTS-17	58.6	55.3	53.4	50.0	55.5	20.3	19.1	18.5	22.0	19.5
LTS-18	52.5	54.3	53.3	58.5	47.8	26.8	27.7	27.0	16.4	25.0
LTS-23	55.4	57.0	55.5	53.7	56.4	23.3	24.0	23.2	21.8	18.8
LTS-25	38.3	37.0	37.6	40.9	37.0	42.8	41.2	41.8	35.7	36.9
LTS-31	58.0	59.0	56.6	59.8	60.7	17.1	17.4	16.8	6.8	12.4
LTS-33	55.9	61.0	58.7	67.6	62.2	17.6	19.2	18.5	4.0	10.4
LTS-41	25.9	25.0	25.6	25.6	23.2	51.9	50.0	53.1	50.0	48.2
LTS-42	32.1	32.3	33.2	29.4	29.7	52.1	52.5	54.7	52.8	52.7
LTS-51	49.5	48.2	48.1	38.7	44.6	36.6	35.6	35.2	43.5	36.3
SBR-15754	52.0	53.1	52.0	...	48.5	25.4	25.9	25.2	...	22.8
SBR-15622	52.4	51.9	51.2	...	52.6	30.9	30.6	29.9	...	24.7
SBR-15497	56.1	61.3	58.8	...	63.6	16.1	17.6	17.0	...	8.9
SBR-15669	30.3	29.6	30.4	...	29.3	57.1	56.0	59.2	...	56.8
LS-1	65.4	69.9	66.9	...	...	17.8	19.0	18.3	...	...
LS-2	70.4	67.3	63.6	...	...	12.0	11.5	11.2	...	...
B-H	56.5	56.9	55.4	...	52.0	24.0	24.1	23.4	...	23.5
B-L	51.7	57.2	56.3	...	55.0	28.9	31.9	31.1	...	25.2

THE SILICATE CONTENTS OF PORTLAND CEMENTS

A detailed discussion of the calibration of the silicate phases was presented in the paper on the combined method (1). The calibration equations obtained in that work apply to the X-ray method as well. The equations relating alite and belite quantities to X-ray relative intensity data are:

$$Aw_2^2 + Bw_2 - \alpha + \frac{R_1}{x_1 + x_2} = 0 \ldots (3)$$

$$Aw_2^2 + (B + CR_3)w_2 - \alpha + \beta R_3 = 0 \ldots (4)$$

in which R_1 and R_3 are the relative intensities of the 1.77 Å alite-belite line to the 3.14 Å silicon line and to the 2.20 Å alite-belite line, respectively; x_1 and x_2 are the weight ratios of alite and belite to silicon, respectively; w_2 is defined by the equation

$$w_2 = \frac{x_2}{x_1 + x_2} \ldots\ldots\ldots\ldots (5)$$

and A, B, C, α, and β are constants having the values -0.0464, 0.1850, -0.0298, 0.1462, and 0.1368, respectively.

Although Eqs 3 and 4 were both obtained in the calibration, only Eq 4 was used in the combined method for the calculation of the alite and belite contents. Equation 4 gave the ratio of alite to belite in a given cement, and again chemical data supplied the additional information necessary for the calculation of the actual quantities of alite and belite. The best values were obtained by using the quantities of silica and alumina available for the two calcium silicate phases.

TABLE III.—SUMS OF THE FOUR MAJOR PHASES IN PORTLAND CEMENTS.

Cement	Oxide Sum, per cent	X-ray Sum, per cent	Combined Sum, per cent	Revised X-ray Sum, per cent	Revised Combined Sum, per cent
1	*2*	*3*	*4*	*5*	*6*
LTS-11........	92.3	99.7	92.1	96.7	92.2
LTS-12........	93.7	91.0	93.6	89.5	93.8
LTS-15........	94.4	97.0	90.7	93.2	90.6
LTS-17........	94.9	91.2	95.3	88.7	95.2
LTS-18........	93.7	96.8	94.4	95.1	94.4
LTS-23........	95.7	97.0	95.2	94.7	95.2
LTS-25........	94.4	92.3	95.1	93.5	95.1
LTS-31........	90.4	90.2	89.1	87.2	89.1
LTS-33........	91.2	95.9	88.8	93.0	88.8
LTS-41........	92.8	90.0	92.6	93.7	92.5
LTS-42........	94.4	96.1	94.3	99.2	94.1
LTS-51........	94.8	94.1	96.0	93.6	96.0
SBR-15754.....	92.0	95.6	92.3	93.7	92.3
SBR-15622.....	94.6	95.5	95.0	94.1	95.1
SBR-15497.....	90.9	95.0	88.4	91.9	88.4
SBR-15669.....	94.4	92.4	94.3	96.4	94.1

The X-ray method makes use of both Eqs 3 and 4. These are solved simultaneously to give values of w_2 and the quantity $x_1 + x_2$. The amount of belite in the cement is then obtained from the equation

$$P_\beta = P_O w_2(x_1 + x_2) \ldots\ldots\ldots (6)$$

where P_β and P_O are the weight fractions (or percentages) of belite and internal standard in the cement. Similarly the quantity of alite in the cement is given by the equation

$$P_\alpha = P_O(1 - w_2)(x_1 - x_2) \ldots\ldots (7)$$

where P_α is the weight fraction (or percentage) of alite in the cement. From these equations, it can be seen that no assumptions concerning alite and belite compositions are employed.

The values of alite obtained by the combined and X-ray methods are given in Table II, columns 2 and 3. A comparison shows that the X-ray values are systematically higher than the combined values by 1.2 per cent, on the average; and the average discrepancy between the two methods is 2.5 per cent. The corresponding values of belite are given in columns 7 and 8. The systematic difference is slight in this case, the X-ray values being 0.2 per cent higher, on the average; and the average discrepancy between the two methods is 1.1 per cent.

THE COMPOSITIONS OF PORTLAND CEMENTS

Because the X-ray and the combined methods give somewhat different values for the four major phases, the question arises, which method gives more accurate results? To test this, the sum of the amounts of the four phases obtained by each method was compared with the sum of the oxides present in the four phases.

Chemical analysis gives the composition of a portland cement in terms of oxides. The four principal oxides are CaO, SiO_2, Al_2O_3 and Fe_2O_3. In addition, for 16 of the 20 cements, analytical data were available for the MgO, SO_3, Na_2O, K_2O, Mn_2O_3, TiO_2 and P_2O_5 contents, as well as for free CaO and insoluble residue. Analytical data for the clinkers were also available.

The calculation of the amounts of the oxides present in the four major phases was carried out as follows.

1. The amounts of alkali sulfates were calculated as proposed by Newkirk (9). The remainder of the alkali was considered to be in the major phases, the remainder of the sulfate in $CaSO_4$.

2. All CaO not in $CaSO_4$ and free lime and all SiO_2 not in the insoluble residue were assigned to the major phases.

3. The amount of MgO in the silicate phases was calculated as in the earlier work (1).

4. All other oxides were assumed to be in the major phases.

The sums of the oxides in the four phases are shown in Table III, column 2. The sums of the four phases obtained from the X-ray method and the combined method are shown in columns 3 and 4, respectively. A comparison of columns 2 and 3 shows that the X-ray method systematically *overestimates* the phases by 1.0 per cent, and the average discrepancy is 2.7 per cent. The combined method systematically *underestimates* the phases by 0.5 per cent, and the average discrepancy is 0.9 per cent.

The conclusion is that the combined method on the average, gives better results than the X-ray method. The combined method underestimates the phases because the minor oxides, with the exception of MgO, were not considered in the phase determinations. The inaccuracy of the X-ray method probably results from the fact that the calibrations were made with pure phases. To be sure, this is true of the combined method, as well. However, in that method, the nature of the determinations compensates, to a considerable extent, for the effects of the impurities.

This can be seen by considering the determination of alite and belite. In the combined method, the only X-ray information used is the ratio of the intensities of two alite-belite lines, R_3 in Eq 4. Because the impurities affect the line intensities in the same direction, their effect, at least partly, cancels out. In the X-ray method, R_1 is also used in Eq 3. This is the ratio of the intensity of a *single* alite-belite line to that of the internal standard line; thus, there is no cancellation of the effects of the impurities.

It has been pointed out that the X-ray method has practical possibilities. It is clear that the method can be improved in accuracy, if the calibration constants for the determination of the four phases are obtained not from the pure phases, but from the phases as they exist in portland cements. This could be accomplished if the amounts of the four phases in a set of portland cements were accurately known. The best approximation to this situation presently available is found in the combined method values of the phases in the 20 portland cements used in these investigations. As the comparison between the combined method sums and the oxide sums indicates, the combined method values, on the average, give reasonably good approximations to the true values.

Earlier in this paper, comparisons were made between the values obtained for the four phases by the two methods. These comparisons showed that the agreement for three of the phases was very good; only alite exhibited significant differences. The recalibration from the portland cement data was, therefore performed only for alite and belite. (Alite and belite cannot be separated.) The new calibration constants, A, B, C, α, and β in Eqs 3 and 4, had the values -0.0864, 0.2335, -0.0536, 0.1596, and 0.1475, respectively.

The recalculated X-ray method values for alite and belite are shown in Table II, columns 4 and 9, respectively. The X-ray method sums of the four phases, using the new alite and belite values, are given in Table III, Column 5. A comparison between columns 2 and 5 in Table III shows that the new X-ray sums exceed the oxide sums by only 0.1 per cent, on the average; and the mean discrepancy is 2.3 per cent.

To find out the effect of the recalibration on the combined method values for alite and belite, these values were also recalculated using the new calibration constants given above. The effects were found to be small. The revised combined sums are shown in Table III, column 6. A comparison with column 4 shows that the original and revised combined sums in no case differ by more than 0.2 per cent.

COMPARISON OF THE FOUR METHODS

In the comparison of the values obtained for the four phases by the two new methods (combined and X-ray) and the two old methods (microscopical and potential compound calculation), the combined method values are used as standards. The combined method is considered the best method available at present for the quantitative determination of the four major phases.

In Table I, columns 4 to 7, the calcium aluminoferrite values obtained by the four methods are given. The average discrepancy between the X-ray and the combined methods is 0.9 per cent, between the microscopical and the combined methods is 2.5 per cent, between the potential compound calculation and the combined method is 0.7 per cent. The microscopical values and the potential compound values given for this phase, as well as for the three other phases, are the same as those given in the earlier paper (1), in which they were discussed in some detail.

In Table I, columns 8 to 11, the tricalcium aluminate values are given. The average discrepancy between the X-ray and combined methods is 0.2 per cent, between the microscopical and the combined methods 1.1 per cent, between the potential compound and the combined method values is 2.2 per cent.

For the alite and belite comparisons, the revised X-ray values are used. In Table II, columns 2, 4, 5, and 6, the tricalcium silicate or alite values obtained by the four methods are shown. The average discrepancy between the X-ray and the combined methods is 1.9 per cent, between the microscopical and the combined methods is 4.6 per cent, between the potential compound calculation and the combined method is 3.6 per cent.

In Table II, columns 7, 9, 10, and 11, the β-dicalcium silicate or belite values are shown. The average discrepancy between the X-ray and the combined methods is 1.0 per cent, between the microscopical and combined methods is 6.0 per cent, between the potential compound and combined method values it is 3.6 per cent.

It follows from the above comparisons that, if the combined method is taken as the standard, the X-ray method gives more accurate determinations of each of the four phases than the microscopical method, and more accurate values for three of the four phases than the potential compound calculation. Although for ferrite, on the average, the potential compound value is slightly more accurate, the X-ray method value will be far more accurate than the potential compound value for ferrites in which the molar Al_2O_3/Fe_2O_3 is markedly different from unity. In view of these considerations, it appears that the X-ray method has possibilities to replace chemical analysis and potential compound calculation in industrial practice.

In conclusion, it may be mentioned that the X-ray method confirms two important conclusions based on the combined method (1). A comparison of columns 2 and 3, Table III, confirms the conclusion that the material called "glassy phase" or "glass" in portland cements is not a true glass, that is, it is not amorphous. Calculation of the Al_2O_3 contents of the ferrite phase and tricalcium aluminate from the values given in Table I, columns 5 and 9, confirms the conclusion that in portland cements not all of the Al_2O_3 is in those two phases;

in other words, part of the Al_2O_3 is in the silicate phases.

SUMMARY AND CONCLUSIONS

1. A new method has been developed for the quantitative determination of the four major phases (tricalcium silicate or alite, beta-dicalcium silicate or belite, tricalcium aluminate and calcium aluminoferrite) in portland cements by X-ray analysis. The method also determines the composition of the calcium aluminoferrite phase. For the portland cements investigated, the composition was close to $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$, on the average.

2. In a method developed earlier by the same investigators, chemical data were combined with X-ray data to evaluate the quantities of the four phases and the composition of the ferrite phase in portland cements. In contrast with this combined method, the X-ray method here reported uses no chemical data and makes no assumptions about the compositions of the phases. A comparison of the two methods shows that the results for three of the phases are in good agreement, and for the fourth, tricalcium silicate, in fair agreement. There is no systematic difference in the tricalcium aluminate results, but for the three other phases the X-ray method gives somewhat higher values.

3. A comparison of the sums of the four phases obtained by the two methods with the sum of the oxides obtained from chemical data shows that the combined method, on the average, is more accurate than the X-ray method. The inaccuracy of the X-ray method probably comes from the fact that the calibration curves were obtained from pure phases, whereas in portland cements the phases are not pure. Because of this, new calibration constants were evaluated for alite and belite, using the data of the combined method as a basis; and the X-ray method values were recalculated, using the new constants.

4. The values obtained for the four phases by the revised X-ray method, by potential compound calculations, and by microscopical analysis, were compared with the values obtained by the combined method, which is the most accurate method available at present. The comparisons show that the X-ray method is more accurate than the two older methods. The results also indicate that the small amounts of impurities present in the various phases do not appreciably affect the X-ray diffraction intensities.

5. The X-ray method has possibilities for increasing application in industrial practice and may gradually tend to replace the classical chemical analysis and potential compound calculation.

Acknowledgments:

The authors wish to express their sincere appreciation to M. A. Swayze, L. T. Brownmiller, Fred Ordway, Katharine Mather, T. C. Powers, and J. B. Alexander for their most helpful comments and suggestions, to L. S. Brown for microscopical examination of the materials used for calibration, to E. E. Pressler for free lime determinations, to Elaine Anderson and Tao-nan Chang for a part of the X-ray analysis, and to I. L. Tyler, T. C. Powers, W. L. De Keyser, A. van Bemst, and M. A. Swayze for providing the portland cements and clinkers used in these investigations.

REFERENCES

(1) L. E. Copeland, Stephen Brunauer, D. L. Kantro, Edith G. Schulz, and C. H. Weise, "Quantitative Determination of the Four Major Phases of Portland Cement by Combined X-ray and Chemical Analysis," *Analytical Chemistry*, Vol. 31, pp. 1521–1530

(1959); PCA Research Dept., *Bulletin No. 108.*

(2) R. H. Bogue, "Calculation of Compounds in Portland Cement," *Industrial Engineering Chemistry (Analytical Edition)*, Vol. 1, pp. 192–197 (1929); PCA Fellowship Paper No. 21.

(3) L. S. Brown, "Long Time Study of Cement Performance in Concrete.—4. Microscopical Study of Clinkers," *Proceedings*, Am. Concrete Inst., Vol. 44, pp. 877–923 (1948); PCA Research Dept., *Bulletin No. 26.*

(4) F. R. McMillan and I. L. Tyler, "Long Time Study of Cement Performance in Concrete. —1. History and Scope," *Proceedings*, Am. Concrete Inst., Vol. 44, pp. 441–453 (1948); PCA Research Dept., *Bulletin No. 26.*

(5) F. R. McMillan and W. C. Hansen, "Long Time Study of Cement Performance in Concrete.—2. Manufacture of the Test Cements," *Proceedings*, Am. Concrete Inst., Vol. 44, pp. 553–601 (1948); PCA Research Dept., *Bulletin No. 26.*

(6) W. Lerch and C. L. Ford, "Long Time Study of Cement Performance in Concrete.—3. Chemical and Physical Tests of the Cements," *Proceedings*, Am. Concrete Inst., Vol. 44, pp. 745–795 (1948); PCA Research Dept., *Bulletin No. 26.*

(7) T. C. Powers and T. L. Brownyard, "Studies of the Physical Properties of Hardened Portland Cement Paste.—4. Studies of Water Fixation," *Proceedings*, Am. Concrete Inst., Vol. 43, pp. 249–336 (1947); PCA Research Dept., *Bulletin No. 22.*

(8) L. E. Copeland and R. H. Bragg, "Preparation of Samples for the Geiger Counter Diffractometer," ASTM BULLETIN, No. 228, Feb. 1958, p. 56 (TP 62); PCA Research Dept., *Bulletin No. 92.*

(9) T. F. Newkirk, "The Alkali Phases in Portland Cement Clinker," *Proceedings*, Third International Symposium on the Chemistry of Cement, Cement and Concrete Assn., London (England), pp. 151–171 (1952); PCA Fellowship Paper No. 63.

CONTROL OF GYPSUM IN PORTLAND CEMENT*

By Bailey Tremper[1]

Synopsis

Progress in devising simple, practical tests to demonstrate the relationship of the SO_3 content of portland cement to its optimum value is discussed. Test methods have been patterned on the principles set forth by Lerch in 1946. These are that for each cement there is an optimum percentage of SO_3 which produces maximum strength and minimum volume change and which is related to the rate of depletion of gypsum during the early stages of setting and hardening. Emphasis is placed on results of cooperative testing for short-time expansion in water and contraction in air of mortars containing the cement in question and the same cement to which pulverized gypsum is added in the laboratory to increase the SO_3 content 0.5 percentage point.

Control of Gypsum in Portland Cement

In 1946, William Lerch (**1**)[2] reported the results of extensive tests on the effect of varying percentages of gypsum in portland cement on the properties of the paste during setting and hardening. Using a conduction calorimeter, he demonstrated that variations in SO_3 produced major differences in the rate of reactions in cement paste during the setting period. These differences affect the hardened paste. For each cement there is an optimum content of gypsum that produces maximum strength, minimum expansion in water, and minimum contraction in air. In many cases the optimum value of SO_3 was found to be higher than the then current specifications would admit.

Lerch also found that the optimum percentage of SO_3 is related to the rate of depletion of the gypsum through interaction with other components of the cement. This was demonstrated by analyses of water extracts of the paste (or mortar) at varying ages. He concluded that when gypsum is present in optimum amount, the water extract contains at least 1.0 g of SO_3 per liter at the age of 18 hr, but less than 0.07 g of SO_3 per liter at the age of 24 hr. The determination of SO_3 in water extracts thus appeared to offer a convenient means of determining the relationship of gypsum content to its optimum value.

Studies by ASTM Working Committee on SO_3

As a result of Lerch's disclosures, ASTM Committee C-1 on Cement organized a Working Committee on SO_3 Content in 1946. Its scope as outlined was to consider all aspects of the effect of SO_3 in portland cement and to devise means whereby gypsum could be controlled by practical tests at its optimum percentage.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Supervising Materials and Research Engineer, California Division of Highways, Sacramento, Calif.; Member, Working Committee on SO_3, ASTM Committee C-1 on Cement.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The first report of the Working Committee was published in the ASTM Bulletin (2). Cooperative testing of clinkers interground with varying amounts of gypsum confirmed the findings of Lerch with respect to an optimum value of SO_3 for greatest strength, minimum expansion in water and minimum contraction in air. Determinations of SO_3 in water extracts of pastes and mortars were made. In general, these extraction, or leach, tests indicated about the same percentage of SO_3 for optimum[3] as was indicated by physical tests of mortars for strength and volume change. Reproducibility between laboratories was not considered to be good enough to warrant a recommendation for specification control of both maximum and minimum percentages of SO_3 by such a test. The Working Committee did, however, recommend deletion of maximum numerical limitations on percentage of SO_3 in specifications for cement and the substitution therefor of a maximum limitation of 0.5 g of $CaSO_4$ (expressed as SO_3) per liter of water extract at the age of 24 hr. It was believed that such a provision would enable manufacturers to proportion gypsum in optimum amount and at the same time would afford protection against an overdose.

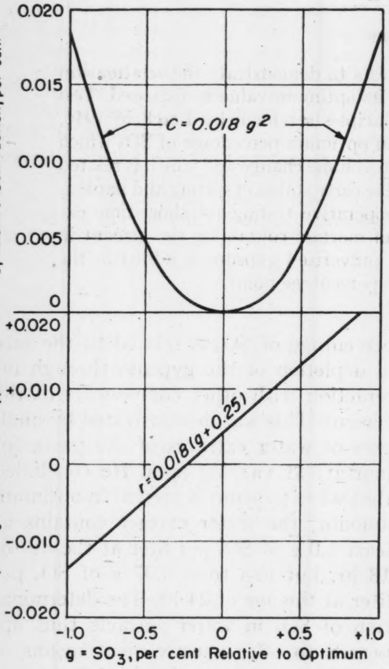


FIG. 1.—Effect of SO_3 on Contraction.

Acting upon this recommendation, the Society in 1952, approved the Tentative Specifications for Portland Cement (C 150 – 52 T),[4] containing such a provision, following the publication of a test method for the determination of calcium sulfate in hydrated portland cement mortar (ASTM Method C 265 – 51 T).[5] The specifications, although apparently permitting sufficient latitude in SO_3 content for optimum results, did not in any way provide assurance that the cement would not contain less than optimum.

Subsequently, the Working Committee conducted another series of cooperative tests for $CaSO_4$ in the water extract. In these tests, the details were prescribed within much narrower limits than in ASTM Method C 265 – 51 and followed very closely the method now designated as ASTM Method C 265 – 58 T.[6] Again the results were disappointing in precision.

In the meantime, a number of producers and consumers had conducted extraction tests and physical tests of mortars. Such data led to the conclusion that the direct specification for maximum SO_3 would achieve the same objective as the requirement for maximum $CaSO_4$ in the hardened mortar, provided limiting values of SO_3 were varied for different types of cement in the light of information then available. In 1956, the Society adopted such specifications and has from time to time made upward revisions of permissible SO_3 in certain types of cement.

---

[3] Based on criteria of more than 0.2 g of SO_3 per liter at 18 hr and less than 0.5 g at 24 hr.

[4] 1952 Book of ASTM Standards, Part 3, p. 4.

[5] 1951 Supplement to 1949 Book of ASTM Standards, Part 3, p. 21.

[6] 1958 Book of ASTM Standards, Part 4, p. 60.

In 1957, the author observed that plots of contraction in air *versus* SO_3 content appeared to produce similar curves for all cements. This suggested the possibility of controlling gypsum by the determination of contraction of the original cement and the same cement to which pulverized gypsum is added to increase the SO_3 content by a fixed amount; 0.5 per cent SO_3 was considered to be a suitable increment.

The suggested method of control can best be explained by reference to Fig. 1.

The upper curve is a parabola conforming to the general equation:

$$C = pg^2 \quad \text{..............} (1)$$

where:

C = contraction in per cent relative to the contraction at optimum SO_3,
g = content of SO_3 in per cent relative to content at optimum, and
p = a coefficient which is dependent on test conditions and characteristics of the cement. For constant test conditions, p is a property of the cement and for convenience in the discussion to follow, is termed the "coefficient of contraction."

In the curve as drawn in Fig. 1, the value of p is 0.018. When tested by the method to be described, experimental results for many cements have been found to agree quite well with this value within the range of -0.5 to about $+0.5$ per cent SO_3 relative to optimum. For higher SO_3 contents, the observed contraction tends to be lower than the equation indicates, possibly because of the restraining effect of unreacted gypsum particles. For some cements the coefficient of contraction is higher or lower than 0.018.

The lower curve of Fig. 1 is a plot of the change in contraction produced by increments of 0.5 per cent of SO_3. It is parallel to the locus of tangents to the parabola which is a straight line and is offset by one half of the test increment of SO_3 or 0.25 per cent. The equation of this curve is:

$$r = p(g + 0.25) \quad \text{..........} (2)$$

where:

$r = dC/dg$ or change in contraction, per cent per unit (1 per cent) of variation in SO_3

For the particular curve plotted in Fig. 1, the equation is:

$$r = 0.018\ (g + 0.25) \quad \text{........} (3)$$

Plots of change in contraction derived from experimental data approximate straight lines in the region of -0.5 to about $+0.5$ per cent SO_3 relative to optimum. For cements containing SO_3 within this range, the determination of change in contraction for an increment of 0.5 per cent in SO_3 yields the data necessary for estimating the relationship to optimum by means of Eq 2. For example, if no change in contraction is produced, the cement contains optimum minus 0.25 per cent SO_3. If the change in contraction is $+0.0045$ per cent, the SO_3 content is optimum for cements having a coefficient of contraction = 0.018. If the change is -0.0045 per cent, the SO_3 content is optimum minus 0.5 per cent if the coefficient of contraction is 0.018. If the departure from optimum exceeds 0.5 per cent, the equation yields less accurate results but nevertheless indicates clearly that the departure is substantial.

This concept was applied in cooperative tests by the Working Committee on SO_3 Content of four cements varying in type, chemical composition and fineness. An "expansion-contraction" test of 1:2 graded Ottawa sand mortar is described in the report[7] of this investigation. In this test, values of contraction are obtained within an elapsed time of 7 days. Interlaboratory reproducibility of the change in contraction produced by an increase of 0.5 per cent in SO_3 was found to be slightly less than 0.005 percentage points. This is interpreted to mean that the test is capable of affording control of SO_3 within 0.25 percentage points of optimum. Values of expansion, obtained concurrently, are useful in providing additional safeguards against an overdose of gypsum.

Concurrently with the expansion and contraction tests, some of the cooperating laboratories made tests for compressive strength in accordance with ASTM Method C 109.[8] The effect of variations in SO_3 content on strength could be evaluated in the same manner as that on contraction. The reproducibility of the strength test however, did not yield as high a degree of discrimination as did the contraction test. This was particularly true for a type II cement which produced a rather flat strength curve.

The study indicated quite conclusively that the percentage of SO_3 that produces minimum contraction also produces maximum strength.

Fragmentary data indicate that 24 hr compressive strength tests of a relatively rich mortar may provide a satisfactory means of controlling SO_3. Because of the reduction in elapsed time, the 24 hr compressive strength test merits further investigation.

TABLE I.—COEFFICIENTS OF CONTRACTION OF TEST CEMENTS.

CALIFORNIA MODIFIED TYPE II, LOW-ALKALI CEMENTS.

Mill	Coefficient of Contraction, *p*
B	0.015
C	0.021
D	0.014
E	0.021
H	0.019
J	0.017
K	0.020 0.014 0.015 0.019 0.014 0.015 Avg 0.016
L	0.017
Average	0.018

REPORT OF WORKING COMMITTEE ON SO_3 CONTENT, OF ASTM COMMITTEE C-1, 1959.

Cement	Coefficient of Contraction, *p*
No. 1	0.011
No. 2	0.015
No. 3	0.015
No. 4	0.013

NOTE.—Coefficients represent rate of change in contraction produced by increment of 0.5 per cent SO_3 when added to the cement containing SO_3 in the range of ±0.25 per cent relative to optimum.

STUDIES BY CALIFORNIA DIVISION OF HIGHWAYS

California Division of Highways has felt concern over wide variations in SO_3 content in cement furnished for its use and the inability to obtain acceptable explanations for such variations.

Acting upon the favorable results obtained in the expansion-contraction tests reported by the Working Committee, the Division of Highways has inaugurated a cooperative series with each of the 13 producing cement mills in the State. The study is confined to modified[9] type II, low-alkali cements meeting the requirements of the specifications of the Division of Highways.

---

[7] Short-Time Tests of Mortars for Controlling SO_3 in Portland Cement at Optimum Value, see Appendix I of Report of Committee C-1, p. 349.

[8] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 58), 1958 Book of ASTM Standards, Part 4, p. 124.

The program consists of two parts. One is the determination by the mill of an appropriate value of p for its cement in Eqs 1 and 2. The second part is the exchange of data of the expansion-contraction test performed by the mill and California Division of Highways using 12 samples provided by the mill at monthly intervals.

TABLE II.—COMPARISON BETWEEN LABORATORIES OF TEST RESULTS FOR CHANGE IN CONTRACTION BASED ON SINGLE TESTS IN EACH LABORATORY.[a]

Mill	Number of Samples	Standard Deviation
A	5	0.0032
C	6	0.0026
D	7	0.0021
E	7	0.0038
F	7	0.0051
H	7	0.0017
I	7	0.0049
J	8	0.0028
Average of mills		0.0037

NOTE.—Comparisons are between laboratory of mill submitting sample and laboratory of California Division of Highways.

[a] Values given are standard deviations of differences in test results expressed in percentage points.

Table I gives values of p as determined by several mills. The coefficient for individual mills was found to vary between 0.014 and 0.021. The mean value is 0.018. The data of the cooperative tests have been analyzed on the assumption that the use of the mean coefficient does not introduce material error into computations of optimum SO_3. Table I also includes values of p that have been computed for the four cements studied by the Working Committee on SO_3 Content.[7]

Interlaboratory results of the expansion-contraction tests that were performed on some of the earlier samples were disappointing but as experience was gained, better agreement was obtained. Table II summarizes the standard deviations obtained for later samples from several mills. The results appear to indicate somewhat better reproducibility for cements of a rather narrow range in composition than was found for the four cements investigated by the Working Committee on SO_3 Content.[7]

Table III presents values of departure from optimum of the cements included in Table II. Departure from optimum was computed by Eq 3. In 85 per cent of the comparisons the difference between laboratories does not exceed 0.25 per cent SO_3. Of the eight comparisons in which this value is exceeded all but two was obtained with cements that were deficient in SO_3 by 0.5 per cent or more relative to optimum.

The results appear to indicate that provided the cement was manufactured to contain SO_3 within 0.25 per cent of optimum as determined by expansion-contraction tests at the mill, the purchaser upon making these tests, would obtain similar results in the majority of cases. Only occasionally would the purchaser obtain results indicating a divergence from optimum as high as 0.50 per cent. In cases of uncertainty, repeat tests by the two laboratories should produce good agreement.

Should the cement contain a substantial overdose of gypsum, contraction data, which may be erratic in such cases, might not disclose the fact. The results of the expansion test are not detailed in this report but examination of the data indicates that excessive values of expansion are reliable signals of excess gypsum.

---

[9] The modification consists of elimination of the requirement for maximum percentage of tricalcium silicate.

Haskell (3) has shown by statistical analysis that optimum SO_3 is closely related linearly to the percentages of alkalies and tricalcium aluminate and the specific surface. He concludes that "any manufacturer could develop a similar linear equation for his own materials and processes." By this means, the manufacturer could control the gypsum within close limits without awaiting the results of expansion-contraction tests. The latter tests would serve to produce confirmatory data and could point to the need of revision from time to time of the equation based on chemical analysis and specific surface.

TABLE III.—COMPARISON BETWEEN LABORATORIES OF INDICATED OPTIMUM SO_3 AS ESTIMATED FROM SINGLE TESTS FOR CHANGE IN CONTRACTION.[a]

Mill	Tested by		Difference Highway – Mill
	Highway[b]	Mill[c]	
A..........	+0.70	+0.50	+0.20
	+0.11	+0.24	−0.13
	+0.09	+0.27	−0.16
	−1.14	−1.31	+0.17
	−0.70	−0.89	+0.19
C..........	+0.16	0.00	+0.16
	+0.22	+0.27	−0.05
	+0.13	+0.05	+0.08
	+0.22	+0.21	+0.01
	−0.78	−0.52	−0.26
	−0.38	−0.38	0.00
D..........	−0.70	−0.61	−0.11
	−0.05	−0.13	+0.08
	−0.75	−0.81	+0.06
	−0.73	−0.57	−0.16
	−0.56	−0.64	+0.08
	−0.54	−0.51	−0.03
	−0.90	−0.70	−0.20
E..........	+0.28	+0.12	+0.16
	−0.61	−0.60	−0.01
	−0.33	−0.50	+0.17
	+0.11	−0.09	+0.20
	+0.09	+0.08	+0.01
	−0.12	−0.12	0.00
	−0.45	−0.23	−0.22
F..........	−0.76	−0.33	−0.43
	−0.48	−0.39	−0.09
	−0.32	−0.72	+0.40
	−0.59	−0.95	+0.36
	−0.47	−0.61	+0.14
	−0.49	−0.48	−0.01
	−0.43	−0.51	+0.08
H..........	+0.23	+0.19	+0.04
	+0.19	+0.31	−0.12
	+0.14	+0.13	+0.02
	+0.29	+0.25	+0.04
	−0.09	+0.05	−0.14
	+0.13	+0.02	+0.11
	−0.03	−0.05	+0.02
I..........	+0.39	+0.41	−0.02
	+0.57	+0.39	+0.18
	+0.28	−0.06	+0.34
	+0.34	+0.38	−0.04
	+0.35	−0.18	+0.53
	+0.33	+0.19	+0.04
	−0.83	−1.13	+0.30
J..........	−0.77	−0.50	−0.27
	−0.32	−0.34	+0.02
	−0.18	−0.15	−0.03
	−0.42	−0.42	0.00
	−0.36	−0.15	−0.21
	−0.11	−0.33	+0.22
	−0.68	−0.60	−0.08
	−0.64	−0.53	−0.11

[a] Values are given in terms of percentage points of excess of deficiency of SO_3 relative to optimum, calculated by Eq 3.

[b] Laboratory of California Division of Highways.

[c] Laboratory of cement mill submitting sample.

CONCLUSIONS

If it may be assumed that the coefficient of contraction of the cements under study does not vary outside the range of 0.014 and 0.022, the test data appear to warrant the following conclusions:

1. For cements of the type specified by the California Division of Highways, it should be feasible for cement manufacturers to control the percentage of SO_3

within ± 0.25 percentage point of optimum by means of the expansion-contraction test and intimate knowledge of materials and processes used.

2. If the SO_3 content of the cement were controlled at the mill within the limits given in (1), other laboratories using the expansion-contraction test should obtain results within these limits in the majority of tests and only occasionally should the indicated deviation be as great as 0.50 per cent. Repeat tests should serve to eliminate or confirm questionable results.

REFERENCES

(1) William Lerch, "The Influence of Gypsum on the Hydration and Properties of Portland Cement Pastes," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1252 (1946).

(2) H. S. Meissner, "The Optimum Gypsum Content of Portland Cement," ASTM BULLETIN, No. 169, Oct., 1950, p. 39 (TP 225).

(3) W. E. Haskell, "Three Factors Govern Optimum Gypsum Content of Cement," *Rock Products*, Vol. 62, No. 4, April, 1959, p. 108.

DISCUSSION

MR. MYRON A. SWAYZE.[1]—The author is to be congratulated for giving us what appears to be a workable measure of what the optimum SO_3 content of any given cement should be. However, I believe it is highly essential that we consider the side effects of such quantities of SO_3 on cement characteristics when the optimum amount is approached. Cement specifications on SO_3 limits have gradually been improved so that we can now approach or reach optimum SO_3 contents in all types of cement. Before such a step is taken, however, the slight decrease in cement shrinkage on drying obtained by optimum SO_3 contents must be balanced against such undesirable characteristics as false set, for example.

Mr. W. C. Hansen's paper on false set, given in Boston a year ago,[2] brought out very clearly that most of the false set we find in cements is due to dehydrated gypsum, and the more of this ingredient we have in cement the greater the tendency for false set. I am sure that if we go to optimum quantities of SO_3 in most cements by increasing the use of gypsum, we will find a pronounced false set in nearly all portland cements. Such a move would be highly unpopular with most cement users who are already plagued with this undesirable property in many cements now on the market.

There is a possible solution for this difficulty, but so far it is not yet practical at many plant locations. This is to control the setting time of cements by moderate additions of gypsum which will be below the level where false set develops, and then make up the difference between this quantity and the level required for optimum SO_3 by additions of anhydrite. To do this of course requires a readily available supply of anhydrite.

In most cases such supplies of anhydrite at present are either not available locally or are not in commercial production. Anhydrite is useless to gypsum producers for manufacture of gypsum products, and they have consequently avoided deposits of gypsum containing anhydrite. There is also a strong reluctance to use anhydrite on the part of many cement plant chemists, which stems from the time when SO_3 specification limits were so low that all the SO_3 in cement had to come from gypsum or quick-set would occur in the cement.

---

[1] Director of Research, Lone Star Cement Corp., New York, N. Y.

[2] W. C. Hansen, "Aeration Cause of False Set in Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 1044 (1958).

To work out such a solution for attainment of optimum SO_3 contents in cement without greatly increasing troubles with false set will require time: first, for gypsum producers to develop reliable supplies of anhydrite for the cement manufacturers and, second, for cement mills to install control equipment to proportion this third ingredient accurately. Time is required for development of both of these changes.

MR. J. S. OFFUTT.[3]—As a member of ASTM Committee C-1, I have watched this development with interest and feel that I can speak for gypsum producers on this subject. They are aware of the need for anhydrite and are working to make it available to cement companies. In the past few years, we have shipped substantial tonnages of anhydrite-gypsum mixtures to a few cement plants. We believe it is possible for the gypsum industry to supply the needs of the cement industry for anhydrite. In some cases, it will be necessary for the cement companies to pay more freight because the closest sources now supplying them gypsum may not have anhydrite. In line with the previous discussion, some of the cement companies have already solved the problem of false set by reducing their grinding temperatures.

MR. T. B. KENNEDY[4] (*for the author*).—The author's paper deals specifically with the use of gypsum to retard cement, but, of course, as Mr. Swayze points out, considerations applicable to cement retarded with gypsum are also applicable to cement retarded with gypsum plus anhydrite. Mr. Swayze points out a real danger of increased trouble with false set with increased SO_3 content, but we are confident this problem can be solved by the cement industry during manufacture. I understand from Mr. Raber that his company is able to overcome the problem of false set.

MR. CHARLES T. RABER.[5]—Some time ago we had a little problem of false set on one of the paving contracts to whom we were supplying the cement.

We had no indication during the manufacturing period that we had any false set in our cement since the hourly average samples going into the silo did not show any signs of false set. This was verified by retesting an average gallon can of the cement about two months later.

When the cement arrived on the project the concrete developed false set or premature stiffening. We tested the subsequent car samples and some of them indicated false set.

In our observations later on, we observed that the cement ground into open bins was free of false set indications and yet the cement stored in 9000-bbl silos had false set tendency after a storage period of one to three weeks or more.

All of our hourly manufactured samples, in 24 average lots, showed no false set whatsoever, and yet when the cement was stored in silos at elevated temperatures we invariably had false set.

Our cement is usually ground in a series of 6 tube mills, open circuit, at a temperature range from 240 to 270 F and then elevated to our storage.

It was my opinion that since we had no false set tendency on the freshly ground hourly samples, which the laboratory cooled immediately after grinding, that it was a matter of temperature, time, and pressure which caused false set in our plant. I proved this in some respect by placing the freshly hot ground cement in 1-gal cans, then into a hot oven (240 to 280 F) for one to three weeks. Some of the hot cement was placed under pressure by means of heavy steel disks. In almost every instance, the cement under pressure had a greater tendency to false set than the loose cement and the longer the storage the greater tendency to false set.

---

[3] Division Merchandise Manager-Industrial Sales, U. S. Gypsum Co., Chicago, Ill.

[4] Chief, Concrete Division, U. S. Army Engineer Waterways Experiment Station, Jackson, Miss.; Chairman, Working Committee on SO_3 Content, ASTM Committee C-1.

[5] Chief Chemist, Giant Portland Cement Co., Egypt, Pa.

This spring we installed a cement cooler, and although our grinding temperatures are about the same we had no false set cement on the freshly ground samples or when the cement was stored in silos for a month or more.

All of our cement is now cooled immediately from an average temperature of 250 F to 110 to 130 F.

It would appear from this experience, before the installation of the coolers, that since there is no apparent tendency to false set on the freshly ground cement even when the cement is ground up to a temperature of 260 F, it does develop false set when stored at elevated temperatures for some period of time. Apparently some of the gypsum is changed to some other form at the prolonged elevated temperatures.

It appears that our cement cooler has also made our cement more free flowing in our open bins.

MR. KENNEDY (*by letter*).—The addition of gypsum, or other forms of calcium sulfate, to cement during manufacture to control rate of hardening is a matter of considerable importance since too little SO_3 results in a product that shrinks too much on drying and too much SO_3 results in excessive expansion and unsoundness.

The importance of the problem is attested to by the fact that a working committee of ASTM Committee C-1 exists to study it.

It is gratifying that the author, who is a member of the working committee, and the cement companies of California are applying knowledge of the relationship between volume stability and SO_3 content to control the amount of gypsum added during manufacture to produce cements for use by the California Division of Highways that will have the best volume stability.

It is hoped that through the interest and efforts of the author and the others on the Working Committee on SO_3 Content of ASTM Committee C-1 that rapid, simple, precise means will soon be perfected for indication of when SO_3 has been added in optimum amount.

With the availability of better test methods it will be feasible to specify SO_3 in a manner that will insure cement and concrete with lower drying-shrinkage and less tendency to crack.

MR. BAILEY TREMPER (*author's closure*).—The discussions deal mainly with problems in avoiding false set in cement should it be necessary to increase the SO_3 content to obtain the optimum value. The major point of concern is a probable increase in manufacturing cost in order to avoid false set under such conditions. Mr. Swayze has questioned that the benefits of optimum SO_3 are commensurate with the cost. It would be futile to attempt a complete discussion of this question without information on what the increased costs might be. Nevertheless there are a number of considerations pointing to a direct saving in cost arising from the use of cement containing optimum SO_3. These are outlined below.

1. Not all cements presently being manufactured require an increase in SO_3 for optimum. Some are approximately at the correct level and others would require a decrease in SO_3.

2. Substantial increases in strength may frequently be obtained if the SO_3 content is raised or lowered as need be to reach optimum.

Cements of higher strength will permit the use of lower cement factors for equal concrete strengths.

3. The author has had occasion to investigate the causes of excessive shrinkage in the exterior concrete walls of a

building which required the expenditure of $100,000 in repairs to prevent rain water from being driven through cracks to the interior. The cement used in construction was high in C_3A and in alkalies. The SO_3 content was far below that now known to be optimum. Had the cement of itself produced less drying shrinkage, it is probable that cracking would have been less s vere and less costly to repair.

4. In a discussion of a paper by Jackson (**1**),[6] it was reported that a concrete pavement some 15 miles in length had suffered deep cracking in a closely spaced pattern following an initial pattern of crazing due to excessive drying shrinkage in the surface. A companion pavement built with the same aggregates but a different cement was entirely free of such defects. The difference in performance must be attributed to a difference in cements. Both contained SO_3 of less than 2.0 per cent. The cement giving poor performance was high in C_3A and alkalies; the other was low in these constituents. The drying shrinkage of the first cement must have been high because of a marked deficiency in SO_3. The SO_3 content of the second cement probably was close to its optimum value. The first pavement required resurfacing over its entire length at a substantial cost.

5. Hveem (**2**) has shown that pavement slabs curl upward at their ends as a result of shrinkage of the upper portion due to unequal loss of moisture. The curled condition is shown to prevail at all times except during warm afternoons when a higher surface temperature tends to flatten the slabs so that they are in contact with the subgrade. Deflections under load applied at the slab end were found to be three times as great when the slab was in a curled condition. Under reduced curling the deflection necessary to bring the slab in contact with the subgrade is less. Higher deflections are not only accompanied by higher stresses in the concrete but they also accentuate pavement pumping and further loss of subgrade support. Cements that produce less shrinkage should reduce curling. This was shown to be the case by profilograph records of the Topeka Test Track (**3**).

Powers (**4**) has stated that the contribution of cement to drying shrinkage of concrete is not one of simple addition to other causes, but is a factor by which all other elements producing shrinkage must be multiplied. Regardless of whatever steps are taken to reduce the drying shrinkage of concrete, none can have its full potential effect unless the shrinkage properties of the cement are also reduced to a minimum.

---

[6] The boldface numbers in parentheses refer to the list of references appended to this discussion.

REFERENCES

(1) F. H. Jackson, "Why Type II Cement," discussion by Bailey Tremper, *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 1225 (1950).

(2) F. N. Hveem, "Slab Warping Affects Pavement Joint Performance," *Proceedings*, Am. Concrete Inst., Vol. 47, p. 797 (1959).

(3) "Profilograph Study—Topeka (Kansas) Test Road," California Division of Highways, Materials and Research Department, July 16, 1956 (unpublished report).

(4) T. C. Powers, "Causes and Control of Volume Change," *Journal of the Research and Development Laboratories*, Portland Cement Assn., Vol. 1, No. 1, Jan. 1959, p. 29.

ACETATE PROCESSED PORTLAND CEMENT*

By B. E. Kester[1]

Synopsis

To overcome the consolidation of dry portland cement that occurs in silos and bulk hopper cars and is commonly termed "pack set," a study was initiated to obtain free-flowing properties of the dry cement. The study culminated in the development of a process, covered by U. S. Patent No. 2,857,286 "Manufacture of Portland Cement."

It was known that certain fatty acids or triglycerides of animal fats would in fact produce free-flowing cement. However, such additives impart water repellency to the cement and entrain air in mortars or concrete. A study of organic acids disclosed that acetic acid had the unique property of creating a free-flowing dry cement and did not, in any measurable manner, alter other properties of the cement. The addition of calcium acetate, either mixed or interground with the cement, was not effective. The reaction of acetic acid with portland cement produces calcium acetate, but since the reaction is dependent upon ionization requiring the presence of water, and water in the vapor state is dispersed throughout the cement, the reaction forms a completely dispersed and surface coating of calcium acetate of the cement grains.

It was further found that soluble salts of acetic acid, which had a greater solubility than calcium acetate, were also effective in creating free-flowing cement.

Acetate salts of lower solubility than calcium acetate are without effect on the free-flowing properties of cement on intergrinding.

With the process for creating free-flowing cement established and with positive data that in other properties the cement was unchanged, attention centered on test procedures to evaluate degree of free-flowing. A simple, qualitative test comparison between treated and untreated cement clearly demonstrates the effectiveness.

For the purposes of this paper, the term "pack set" is used and described as the tendency of cement to cling to itself and to become more resistant to free flow with time and vibration. This phenomenon, exhibited by many finely ground powders, is in no way related to hydration of cement in storage, or to surface hydration of sacked cement, known as "warehouse set."

This common ailment of "pack set" was aggravated beyond tolerable limits by the circumstance of a particular field use of the cement. In the oil fields of Illinois, type III portland cement is used for oil well sealing grouts. Beyond the normal problems of silo withdrawal of this high-fineness, high-early-strength cement, cement shipped in bulk hopper cars went through several switchyard transfers. Each transfer further consolidated the cement and enhanced "pack set." Car unloading became a major problem for the user. After the usual difficulty of removing cement from the hopper car and then from the small field bulk silos, it was loaded into bulk cement trucks. Most of these trucks discharge their cargo by an integral screw. After bouncing on the back gravel roads of Illinois for from 20 to 80 miles, the cement had again consolidated to the point where great difficulties were experienced in unloading from the truck at the actual job site.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Technical Director, Missouri Portland Cement Co., St. Louis, Mo.

It has been observed by many producers that this condition of "pack set" is not constant and that certain cements exhibit more setting than others and, that at times cements from a single source have a greater or lesser tendency to "pack set." It is believed that the phenomenon of "pack set" is a surface energy characteristic and might well be expected to vary with variations in manufacturing processes or changes in ambient conditions. By experimental plant scale tests, it has been fairly well established that cements ground from clinkers with all kiln dust returned from an electrostatic precipitator have a greater tendency toward "pack set" than cements ground from clinkers with the kiln dust discarded. The reasons for this variation, while of interest, are beyond the scope of this paper.

It has been frequently observed that "pack set" tendencies become more severe in the fall of the year. From these observations, it has been concluded that the minimum inventories of the fall and the practice of kiln to mill production, followed by shipment as soon as early tests are completed, resulted in the so-called "green cement," which is more apt to "pack set" than normal.

REDUCTION OF "PACK SET" BY ADDITIVES

Prior to World War II, the company had produced a special cement for block plant producers which had excellent free-flow properties and which exhibited no tendency to "pack set," despite imposed conditions of time, temperature, and vibration. The free-flowing nature of this cement was created by the addition of 0.02 per cent yellow grease, a crude packing house product which is primarily a mixture of triglycerides of hog fat. Unfortunately, the yellow grease also imparted water repellancy and entrained large quantities of air. Both of these characteristics were undesirable for the intended use of the type III cement.

Other investigators (**1**)[2] had shown that portland cements which had been made water repellant, and subsequently free flowing and unlikely to "pack set," would, if the entrained air could be suppressed, have physical properties similar to untreated cements both in mortars and concrete. This is demonstrated by the so called "hydrophobic" cements based on either intergrinding or intermixing of oleic acid (for water repellancy) and tri-*n*-butyl phosphate (for air detraining).

This process, while of considerable value in inhibiting "warehouse set," and undoubtedly successful in reducing "pack set," involves additional costs in the order of $0.10 per barrel, which is not economically justified.

However, these studies prompted a study of the influence of a number of organic acids, including acetic acid, on properties of portland cement. It was apparent from laboratory millings that acetic acid when interground with cement clinker and gypsum at commonly prevailing mill temperatures created a free-flowing cement with no indicated water repellancy and no entrainment of air.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The first concern was what property of cement, other than the flowing characteristics of the dry powder, had been changed by the acetate treatment. In the approximate eight years that the process has been actively practiced, no single property of cement other than its flowing characteristic has been found to be altered. Table I shows comparative analyses of treated and untreated cement which are typical of many such analyses made over the years.

TABLE I.—PHYSICAL TESTS—TYPE III ACETATE-PROCESSED PORTLAND CEMENT—ASTM SPECIFICATION C 150 - 56.

Physical Properties	Average Value of 4 Test Samples—Additive: Glacial Acetic Acid		
	None	0.02 per cent	0.10 per cent
Fineness, specific surface, sq cm per g:			
Air permeability test	4920	4945	4930
Turbidimeter test	2610	2600	2615
Time of setting Gillmore test:			
Initial set, hr:min	2:17	2:15	2:15
Final set, hr:min	4:18	4:20	4:15
Air content of mortar, per cent by volume	6.3	6.3	6.3
Soundness:			
Autoclave expansion, per cent	0.05	0.06	0.05
Tensile strength, psi:			
1 day	346	345	340
3 day	430	420	440
7 day	480	485	500
28 day	560	570	570
Compressive strength, psi:			
1 day	2925	2900	2910
3 day	4440	4400	4390
7 day	5370	5350	5395
28 day	6520	6550	6490

The test results shown in Table I are based on plant-produced cements from successive mill operation periods, using no additive during the first portion of the production period and acetic acid in two successive additive amounts.

A composite test sample, taken from 15-min grab samples, was prepared from each portion of the above production periods of each condition. The three composite samples were, after thorough blending, each subdivided into four test samples. The reported results are the average of all individual tests made on the four test samples, which represented each of the three mill operation periods of 8-hr duration.

Additional tests of freezing-and-thawing resistance, efflorescence tests, concrete tests with locally available gravels and crushed stones, with Meramec sand, and with river sand contaminated with lignite fail to disclose any detectable performance differences. When suitable air entraining agents are added to the concrete mix, the acetate-processed cement will entrain air equally as well, but to no greater degree than untreated cement.

Since it was established that the properties of acetate-processed cement, other than its flow characteristics, have been unchanged, production of the treated cement was started and shipments made to the oil fields. During an 8-yr period, no single complaint on bulk hopper car unloading, bulk truck difficulty, or any complaint on any cement property has been received from the users, with one exception. It is difficult rapidly to evaluate the tendency to "pack set," and on one occasion when the acid pump had broken down, cement produced during this period which looked to be free flowing, was shipped. A few hours after a bulk hopper car had arrived at the oil fields, a complaint arrived at the plant that the cement was "sticky." The silo of insufficiently-treated cement was circulated by means of a cement pump and acetic acid introduced to the pump hopper in proper proportions. Subsequent cars shipped from this silo were wholly acceptable.

Since the earliest use of acetic acid, it has been introduced into the tube mill feed trunnion by a simple positive displacement pump through a $\frac{1}{4}$-in. pipe at a rate from 0.02 to 0.05 per cent by weight, based on total dry solids. In view of early field successes of the treated cement, there was little concern either with the search for other materials which might prove effective, or with measurement of degree of effectiveness. The problem had originally been a program of necessity and acetic acid had fulfilled the need; the process became standard practice without further study. A patent application had been filed; the customers were satisfied and the process was simple and inexpensive.

The United States Patent Office, in citing interferences however, brought attention back to the basic nature of the problem and acquired some refinement of the knowledge. A previous patent had issued covering calcium acetate as a cement additive.[3]

The effect of calcium acetate on the properties of portland-cement clinker and of portland cement has been previously reported by Juniper and Kalousek (2,3), who found a definite retardation and subsequent reduction in compressive strength test results caused by the addition of calcium acetate. This work, however, was based on rather large increments of calcium acetate with per cent of acetate radical in the order of 1.0. The study was also confined to the effect of calcium acetate interground or intermixed with portland-cement clinker rather than normally retarded portland cement containing additive gypsum.

In the case of minor amounts of acetate radical addition to an otherwise normal portland cement, no change is noted at early ages of test, but a definite major increase in compressive strength was shown at 1 and 2 yr age.[4]

A brief study of calcium acetate with laboratory mill trials proved it to be ineffective in creating a free-flowing cement. While calcium acetate produces the free-flowing effect, a markedly different situation prevails when acetic acid is interground with cement than where calcium acetate is interground. That calcium acetate is the active effective component can be visualized by the realization that in the presence of moisture vapor with attendant ionization, the reaction is as follows:

$$\mathrm{CaO} + \mathrm{H_2O} \cdots \mathrm{Ca(OH)_2} + 2\ \mathrm{CH_3COOH} \cdots \mathrm{Ca(CH_3COO)_2 \cdot 2\ H_2O}$$

By adding acetic acid to the milling operation, the reaction is not immediate since it requires the presence of water to ionize both the acid and the calcium oxide. What water is present in the cement powder is extremely dispersed and is present from the prevailing relative humidity of the surrounding air. The net result of these conditions is the creation of calcium acetate throughout the mass of cement and individually surrounding each cement particle. This is a markedly different situation than that which prevails in intergrinding calcium acetate with the cement. In this latter case, the dispersion of calcium acetate would be entirely dependent upon how fine the salt could be ground and dispersed throughout the cement. This fineness and dispersion could in no manner approach the degree of dispersion of water and acid vapor which prevails on injection of acetic acid in a grinding system.

Prior art patents for the use of fatty

---

[3] U. S. Patent No. 346,525, James Anderson, Aug. 3, 1886.
U. S. Patent No. 747,689, John Joseph Feely, Dec. 22, 1903.

[4] H. L. Vanderwerp, Technical Director, Peerless Cement Co., personal correspondence with the author.

acids such as stearic, oleic, palmitic, butyric, and others were evaluated.[5] It has been successfully demonstrated that acetic acid is unique in that all fatty acids either entrain air or impart water repellancy. The film formed of calcium acetate does not repell or retard water-wetting of the cement grains. In fact, it in no way alters the mixing characteristics of the cement in either neat slurries, mortar, or concrete.

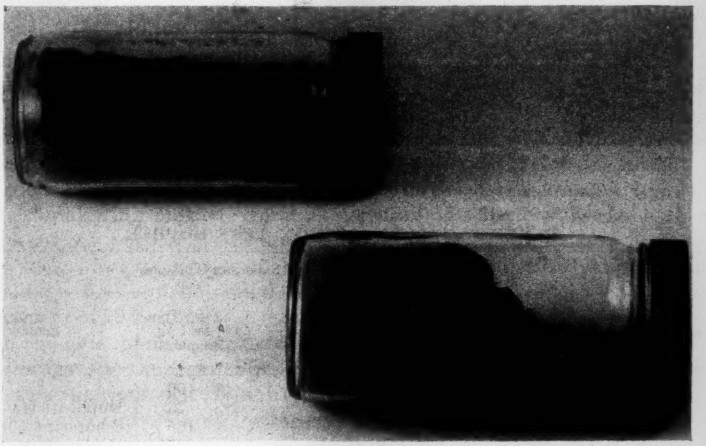


FIG. 1.—Qualitative Measurement of Flow Characteristics of Treated and Untreated Portland Cement.

It has been demonstrated that the use of soluble salts of acetic acid is effective if the proposed salt has a greater solubility than calcium acetate. In such a case, the laws of mass action cause a reaction to proceed toward the formation of calcium acetate. Since again this reaction can only proceed in the presence of ionization, which requires the presence of water, and the only water present in the cement is in a completely dispersed water vapor state, the net result will again be the formation of calcium acetate throughout the mass and individually surrounding each cement particle.

A typical example of a soluble acetate salt is ammonium acetate which has a solubility of 140 g per 100 ml water compared to calcium acetate with a solubility of 37.4 g per 100 ml. The reaction then proceeds according to the following equation:

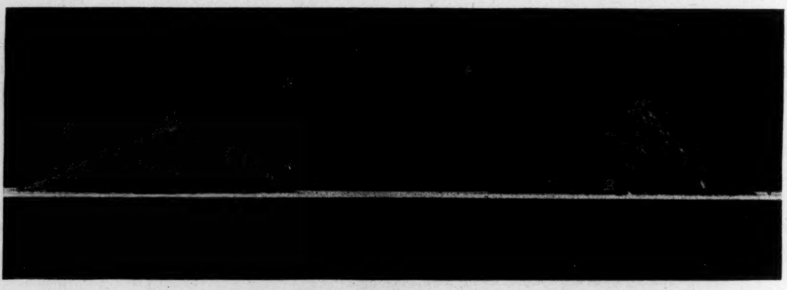
$$CaO + H_2O \cdots Ca(OH)_2 + 2\ CH_3COONH_4$$
$$+ H_2O \cdots CA(CH_3COO)_2 \cdot H_2O + 2\,NH_4OH$$

A number of acetate salts having greater solubility than calcium acetate were interground on a laboratory ball mill under temperature conditions commonly prevalent in actual production, or approximately 200 F. When interground on the basis of per cent weight of the acetate radical, ammonium acetate, aluminum acetate, cobalt acetate, ferrous acetate, sodium acetate, and potassium acetate were all equally effective in developing the free-flowing property.

---

[5] U. S. Patent No. 1,640,602, H. Ditlinger, Aug. 30, 1927.
U. S. Patent No. 1,979,380, H. A. Gardner, Nov. 6, 1934.
U. S. Patent No. 1,947,504, H. R. Starke, Feb. 20, 1934.
U. S. Patent No. 1,957,415, E. J. Wechter, May 1, 1934.

A similar test series based on acetate salts less soluble than calcium acetate was run. Intergrinds were made with copper acetate, chromium acetate, bismuth acetate, and ferric acetate added on the same basis of weight per cent of the acetate radical and were without effect on the flow properties of the resultant cement.



Courtesy of Rock Products Chemicals Division, W. R. Grace & Co., Dewey and Almy Division.

FIG. 2.—Angle of Repose of Treated *versus* Untreated Portland Cement.

TABLE II.—FLOW RATE ON TEST HOPPER.

Type I Portland Cement—Mill and Laboratory Samples

Sample	Source	Additive	Flow Time for 2 kg Sample, sec	Remarks
A...........	Mill sample	None	100	....
A...........	Mill sample	None	73	Duplicate test
B...........	Market sample	None	165	Complaint sample
B...........	Market sample	None	125	Duplicate test
C...........	Market sample	None	74	Acceptable
C...........	Market sample	None	64	Duplicate test
D...........	Market sample	None	164	Complaint
E...........	Mill sample	None	88	....
E...........	Mill sample	None	70	Duplicate test
F...........	Laboratory ball mill	None	37	....
G...........	Laboratory ball mill	None	64	Stored 24 hr hot
H...........	Laboratory ball mill	0.02 per cent glacial acetic acid	5	Interground
I...........	Laboratory ball mill	0.10 per cent ammonium acetate	2	Interground
J...........	Laboratory ball mill	0.50 per cent water	88	Interground

LABORATORY EVALUATIONS

With the use of the acetate process largely based on visual evaluations of the differences between treated and untreated cements, substantiated by the elimination of "pack set" in storage silos, rail bulk hopper cars, and in the field bulk trucks, some attention was given to a simple laboratory evaluation of the flow characteristics of the cement. Two 8-oz glass jars were partially filled with the test cement and an untreated cement of the same type. These were consolidated for 60 sec by tapping the jar on a rubber padded table top. The consolidated samples were then rolled across the top of a desk, and the distance of rolling required to level the cement in the jar was taken as a rough estimate of flowability. Figure

1 shows the differences between treated and untreated cements at the same distance of rolling. In the course of four turns of the jar, the untreated cement (*a*) has just started to flow, while the treated cement (*b*) has become nearly level in the jar. This crude qualitative test served as a rapid check on the acetate process treatment and was adequate to insure delivery of free-flowing cement to the customer.

Considerable industry interest has been aroused and has required that a more quantitative measure of the effect of acetic acid be available to evaluate the process on a wide variety of cements.

Replacement of the hand tapping of the sample container with consolidation by means of an electric vibrator gives more reproducible results; small laboratory model pulsating magnet vibrators serve well for this purpose. With the use of such consolidation, a normal ratio of flowability between untreated and treated cement of 20 to 1 has been experienced. In one series of tests conducted by other investigators[6] the treated and untreated cements were placed in Erlenmeyer flasks and consolidated by 15-sec vibration. The flask was then supported in an horizontal turning rack and the greater the number of rotations required to cause the packed cement to slough from the bottom of the flask, the greater the "pack set" tendency of the cement.

This is a refinement of the original technique and lends itself to numerical expression as follows:

Cements	Number of Rotations after 15-sec Vibration
Blank	40
Acetic acid treated	2
Ratio, blank to acetic acid treated	20 to 1

Figure 2 shows the difference between untreated and treated cements by yet another means. In this case, a 4-in. glass funnel is filled with the test cement. The funnel is attached to an electric vibrator through means of a funnel rack. Here, two distinct numerical values can be obtained. The angle of repose of the two samples can be compared. In Fig. 2(*a*), the treated cement has a 28-deg angle of repose, while the untreated cement (*b*) has a 45-deg angle of repose. Also, the time for passing a given weight of cement through the vibrating funnel can be numerically expressed as a measure of flowability.

A fourth method proposed consists of measuring the time required to pass a 2000-g sample of dry cement through a small test hopper, under standardized conditions. The rectangular hopper is 9 by 9 in. at the top and 9½ in. high. The opening at the bottom is 1½ by 1½ in. and is fitted with a hinged door and latch for manual release. The top of the hopper is crossed by a 1 by ¼-in. flat iron strip. A 1 by 1 by 2¼-in. steel rapper strikes this crossbar at its mid-point. The rapper has a stroke of 1½-in. and drops 18 times per min. It is lifted by a 19½ by ½-in. camshaft; the motor, speed reducer, and cam being located on the base plate of the assembly.

The flow time is the average time (in seconds) required to pass a 2000-kg sample of cement through the hopper. An average of ten consecutive passes is used for reported value.

The results shown in Table II indicate the same order of flow increase for treated cements as shown under the rotating Erlenmeyer flask.

CONCLUSIONS

While testing procedures are still crude and empirical, it is apparent that the increased flowability of acetate-processed cement is not only well founded by field experiences but can be demonstrated by a number of laboratory techniques. No other detectable difference, either in field use or laboratory evaluations, has been noted.

---

[6] Rock Products Chemicals Division, W. R. Grace & Co., Dewey and Almy Division, Cambridge, Mass.

REFERENCES

(1) U. W. Stoll, "Hydrophobic Cement," presented at the Second ASTM Pacific Area National Meeting, Los Angeles, Calif., Sept. 16–21, 1956 (*not published*).

(2) Charles H. Juniper and George Kalousek, "Effect of Admixtures on Portland Cement," *Rock Products*, Vol. 45, No. 4, April 1942; Vol. 45, No. 5, May 1942.

(3) E. S. Newman, R. L. Blaine, C. H. Juniper, and G. L. Kalousek, "Effects of Added Materials on Some Properties of Hydrating Portland Cement Clinkers," *Journal of Research*, National Bureau of Standards, Vol. 30, No. 4, April 1943.

DISCUSSION

MR. F. J. MARDULIER.[1]—I believe that the author has been much too modest in disclaiming any research capabilities in connection with the work undertaken at his plant over the last years in connection with the "pack set" development. The fact is that he has developed a practical solution of a problem which is important to all producers of finely ground powders.

The author was also too complimentary of our efforts in this connection when he attributed to us the development of the Erlenmeyer flask approach for the quantitative measurement of "pack set." Actually, this idea was originated by the Louisville Cement Co. At least it is they from whom we obtained it. We proceeded to speed the test up a bit by substituting a magnetic vibrational table for the device they had been using for compaction. We also took certain steps to refine it, to improve its reproducibility. This was a normal process of refinement undertaken by the head of our physical testing laboratory. The test, however, does have the merit of giving quantitative figures which we feel are reasonably accurate measurements of "pack set."

I would like to stress a point which the author has already mentioned but which I believe from my conversation with other people deserves a little emphasis. In the problem of nonflowing or sticky cement there appear to be two facets. One is the resistance of that cement to starting to flow, the other is the rate at which that cement flows once flow has begun. I believe this is analogous to a comparison of starting friction to moving friction. Just as starting friction is always greater; "pack set" which is the term applied to the resistance of cement to start flowing is also always greater. We have emphasized the problem which seems to be more basic, namely, the apparent "set" of cement which has been compacted.

The fluidity test as described by the rolling bottle is a measure of the other facet of sticky cement, namely, its fluidity after flow has started. Likewise the comparison of the two piles of cement with their different angles of repose is another indication of cement fluidity or flowability. We agree with the author that flow ability and "pack set" are interrelated.

MR. B. E. KESTER (*author*).—I would like to make one comment and I should have made this earlier. The "pack set" phenomenon as relates to hopper cars, silos, etc., of course, deals with this initial starting of flow and the flow rate itself may or may not have any significance on continuing the emptying after flow has begun. However, I am informed by the people who make air conveying equipment that there are cement plants where they will not even recommend the installation of air conveyors because the cements have such poor fluidity. And here we are dealing with the true phenomenon of cement fluidity, and I might say in those plants where this condition has been observed, they are interested in the phenomemon of fluidity, *per se*, not "pack set."

---

[1] Manager, Rock Products Chemicals Department, Dewey & Almy Chemical Division, W. R. Grace & Co., Cambridge, Mass.

MR. W. C. HANSEN.[2]—I think the author is to be complimented on this fine piece of work. You indicated that salts with greater solubility than calcium acetate were equivalent, maybe you did not use the word "equivalent," to acetic acid. I wonder if that is true or whether the acetate ion in acetic acid is the most effective ion and that the effect decreases as the solubility of the salt approaches the solubility of calcium acetate.

I would also like to ask what concentration of acetic acid is used when it is used in the mill?

MR. KESTER.—The effectiveness of salts is based upon the acetate radical, and all that we require in actual operation is that that acetate radical of the salts be of greater solubility than calcium acetate. This is so that the formation proceeds toward formation of calcium acetate surrounding each individual grain. Now the additive amounts are in the order of 0.02 per cent of the acetate radical, and this would of course imply that if you were using ammonium acetate you would be using 0.027 per cent. Whatever the molecular weight of any acid salts that you were using would be proportional to 0.02 per cent acetic acid. The material is effective without deleterious effect upon the cement over the range from 0.01 of 1 per cent, to about 0.2 of 1 per cent. We have never actually produced commercial cement with higher than 0.2 of 1 per cent additive acetate.

MR. H. L. VANDERWERP.[3]—I would like to suggest that there may be one other facet to the addition of acetic acid favorable to this process.

Several years ago we added, not acetic acid, but calcium acetate, to portland cements in small amounts and finding as you did that there was no difference in the physical tests at the early ages. Surprisingly, however, at the ages of 1 yr and more, we found increases in strength of considerable amounts.

I have never had a chance to pursue it further but I have been interested in it.

MR. KESTER.—I must apologize to Mr. Vanderwerp and to this group, we do not have long-time test data on acetate processed cement. We had assumed from test data including 90-day strength values that the acetate process did not alter physical characteristics of either mortars or concrete. I am sorry we do not have your correspondence and test data.

MR. W. J. MCCOY.[4]—I have one question to ask regarding ammonium acetate.

Have you ever used this material in a plant grind? It would be convenient to handle since it is a solid but ammonium salts have that peculiar characteristic of releasing ammonia fumes when they are in the presence of alkali. As would be expected this action is accelerated by heat. Even though the quantity of ammonium acetate used is very small you might still have a considerable volume of ammonia gas given off.

MR. KESTER.—We did make an experimental grind with ammonium acetate. Mr. McCoy is quite correct; the ammonium fumes are not particularly pleasant. However, with close circuit grinding systems, the fumes are largely exhausted through the dust collectors following the air separator and in most cases would not be noticed by either plant personnel or neighbors.

---

[2] Director, Research Laboratory, Universal Atlas Cement Division of U. S. Steel Corp., Gary, Ind.

[3] Technical Director, Peerless Cement Co., Division of American Cement Corp., Detroit, Mich.

[4] Director of Research, Lehigh Portland Cement Co., Coplay, Pa.

STATISTICAL ASPECTS OF THE CEMENT TESTING PROGRAM*

By W. J. Youden[1]

Synopsis

A statistical technique has been developed for increasing the amount of information obtained in interlaboratory test programs. The main feature of the technique consists in a graphical presentation of the results reported for pairs of samples. The graphs make for easy identification of laboratory bias and provide a method for estimating the precision of the test procedures.

A rating system has been established to facilitate the examination of a large mass of data. The theoretical distribution of average scores was computed in order to provide a yardstick of judgment for the observed distribution of average scores.

When a reference sample is tested by a considerable number of laboratories, one consequence is to make it possible for each laboratory to find out how its own result compares with the results obtained by all the other laboratories. Unfortunately, relatively little can be learned from the collection of results reported for one reference standard. Examination of the results usually shows one or more absurd results that presumably are caused by slips in computing, typing, or misreading of an instrument. Generally these extreme values are fairly obvious and may safely be set aside. The next step is to obtain the average of all the remaining results and perhaps some measure of the scatter or error.

It should be remembered that a collection of results from different laboratories is not the same as a collection of repeat tests on one material by one laboratory. In the latter case any systematic error that may be associated with the laboratory's technique for conducting the test will be present in every result as a "constant" error. The observed scatter in these repeat determinations arises from *random* errors and reflects the *precision* of that laboratory. Any one result from that laboratory is a composite of the unknown true value, μ, the unknown constant error for that laboratory, L_i, and a random error e_i.

A collection of results, one from each of n laboratories testing the same material, will have the following composition.

$$\begin{array}{c} \mu + L_1 + e_1 \\ \mu + L_2 + e_2 \\ \vdots \\ \mu + L_n + e_n \end{array}$$

The scatter exhibited by these n quantities may arise from a complex of circumstances. Each laboratory will have its own systematic error, L. In addition the precision may vary from laboratory to laboratory so that each random error comes from a different normal distribution. Given a collection of results, one from each laboratory, there is no way whatever to unravel this complex of circumstances. Given a parallel set of results on a second sample, some progress can be made, if certain assumptions are made. These assumptions are (1) that each laboratory conveys its particular systematic error equally to both materials and (2) that for all practical purposes, differences in precision among the laboratories can be ignored.

---

* Presented at a special session on Statistical Treatment of Interlaboratory Test Results, Including a New Graphical Method sponsored by Committee C-1 on Cement held on June 25, 1959 during the Sixty-second Annual Meeting of the Society.

[1] National Bureau of Standards, Washington, D. C.

By definition a systematic or "constant" error should persist in all results made by the laboratory, provided the materials are fairly similar and do not differ too much in the magnitude of the property tested. If these conditions do not hold and the systematic error varies with the type of material, the complications multiply. There is abundant evidence in the results reported by Blaine and Crandall (**1**)[2] that systematic errors do vary from laboratory to laboratory and that the systematic error of a laboratory is conveyed to a series of materials. Even more important the evidence is conclusive that when a laboratory does depart markedly from the concensus of the other laboratories, the systematic error is responsible in most cases. The identification of systematic errors as the major cause of divergent results makes the role of precision relatively minor. Perhaps this is fortunate because the detection of even substantial differences in precision between two laboratories requires a score or more of repeat determinations from each laboratory.

The collection of results, one from each of *n* laboratories, reflects a collection of *n* systematic errors, some small, some large. The magnitudes of these systematic errors may also be distributed in a manner approximating the normal. The fact that the gross results (which include both systematic and random errors) usually approximate the normal distributions supports this view. It is not surprising that among the systematic errors there may be a few exceptionally large ones. The results with large systematic errors contribute a substantial, sometimes even a major, portion of the sum of the squared deviations used in calculating the standard deviation. This inflation in the sum of the squared deviations gives a value for the standard deviation that is larger than it should be, larger than the one actually being achieved by possibly 95 per cent of the laboratories.

There would be some justification in resigning oneself to accepting this overlarge estimate of the standard deviation if the divergent results arose from chance or random errors. In that event, these large deviations would, in repeat studies, turn up from different laboratories. The study with a dozen cements shows this not to be the case. The same laboratories are identified with the large deviations, the sign of the deviation remaining the same. As soon as certain laboratories discover that they have, somehow, acquired rather large systematic errors, the way is open for corrective measures to be taken. In fairness to the test procedure, any appraisal of its merits should not include the work of laboratories that reflect persistent large systematic errors because these greatly inflate the estimate of the standard deviation. Finally, any progress toward an over-all improvement in testing is most easily achieved by a few laboratories taking corrective action.

THE PROBLEM OF DIFFERENTIATING AMONG LABORATORIES

Suppose we have 100 laboratories, all without systematic errors, that is, only

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

random or precision type errors. First imagine that all have the same precision in conducting the three-day tensile strength test. If the tensile strength is taken as 440 psi and the standard deviation for all the laboratories is 40 psi, it is an easy matter to calculate the expected distribution of the test results. Statistical tables show that four laboratories may be high by 70 psi or more, and four laboratories low by 70 or more psi.

TABLE I.—PREDICTED DISTRIBUTION OF RESULTS REPORTED BY 100 LABORATORIES. ASSUMPTION A: ALL LABORATORIES HAVE STANDARD DEVIATION OF 40 PSI. ASSUMPTION B: ONE-THIRD OF THE LABORATORIES HAVE STANDARD DEVIATION OF 30; ONE-THIRD A STANDARD DEVIATION OF 40; AND ONE-THIRD A STANDARD DEVIATION OF 50.

Tensile Strength, psi	Predicted Number of Laboratories	
	Assumption A	Assumption B
510.0 and more	4.0	4.3
490.0–509.9	6.5	6.1
470.0–489.9	12.1	11.6
450.0–469.9	17.5	17.8
430.0–449.9	19.8	20.5
410.0–429.9	17.5	17.8
390.0–409.9	12.1	11.6
370.0–389.9	6.5	6.1
less than 370.0	4.0	4.3
	100.0	100.1

The second column in Table I shows the expected number of laboratories obtaining results in various ranges of breaking strengths. When the results are reported back to the laboratories, those close to the average will undoubtedly feel rather pleased. No grounds whatever exist for this self-congratulation because repeat tests will find the participating laboratories shifting up and down the scale in a chance manner. Remember that the table was calculated on the assumption that *all* the laboratories really have the same precision (and systematic errors were absent.)

Now imagine an alternative situation whereby one third of the laboratories have a standard deviation of 30 psi, another third a standard deviation of 40 psi, and the remaining third a standard deviation of 50 psi. Again the expected distribution of laboratories can be calculated and it is clear from the third column in Table I that the scatter of the results is more or less indistinguishable from that found when all the laboratories had a standard deviation of 40 psi. But it may be noted that under assumption B there is a slightly greater concentration of results in the vicinity of the mode, and in the extreme "tails," with corresponding deficiencies at moderate deviations. Such greater concentration near the mode, balanced by larger "tails," is a characteristic of distributions composed of results of unequal precision, and in practical work may usually be taken as an indication of the presence of results of unequal reliability. Unfortunately, there is no trustworthy way of identifying in such a distribution of single results which correspond to the higher precisions and which to the lower. Laboratories close to the average may be happy about it, but no way exists to justify their satisfaction using only this one set of results.

If repeated test results are available, then the way is opened to differentiate among the groups. Ultimately membership in one of the 30, 40, and 50 psi standard deviation categories could be established. But even if as many as 25 repetitions of the test are run by each laboratory, the discrimination is rather poor. For example, one out of five of the 40 psi standard deviation laboratories will be credited with a standard deviation of 35 or less and hence be classified as belonging to the 30 psi group. And a second one of the five will have the bad luck to have its estimate be 45 or more and be put down as belonging to the 50 psi group. Discrimination among laboratories as to precision is not easy. On the other hand, constant errors very soon reveal themselves. Thus, a constant error equal to the standard deviation will lead to a preponderance of deviations of the same sign, the expected proportion being 0.84.

THE TWO-SAMPLE PROGRAM

The prevailing presence of systematic errors in test results can be demonstrated if a number of laboratories perform only one test on each of two test materials. A simple graphical representation (2) of the *n* pairs of results obtained from the *n* participating laboratories suffices to show that systematic errors are present. On a piece of graph paper draw the usual *x* and *y* axes. On the *x* axis lay off a scale of values covering the range of values found for one of the materials. Lay off a similar scale using the same unit on the *y* axis. The pair of values for the two materials reported by a laboratory may now be used as coordinates to determine a point representing the work of that laboratory. A point is plotted for each laboratory. The collection of points is then divided by a horizontal line so that half the points are above the line and half below. A vertical line is also drawn dividing the points half to the left of the line and half to the right. These two lines partition the graph paper into four quadrants. The upper right quadrant corresponds to a region where points represent values greater than the median for both materials. The lower left quadrant is a region where the points represent values less than the median for both materials. The other two quadrants provide for values, one of which is greater than the median and one below the median. The two quadrants correspond to the two ways this can happen.

The median is less disturbed by extreme values and is convenient because counting is quicker than computing the averages. The intersecting lines may be drawn through the point determined by the average for each material if that is preferred. Usually this makes an imperceptible difference in the location of the lines. When there is a difference in location, it comes about from the inclusion of one or more "wild" values in the averages and the median values are preferable.

Results greater than the median are considered to give plus deviations and results less than the median are taken to give negative deviations. The four quadrants thus are identified with the four possible paired combinations (++, +−, −+, −−) of deviations from the averages. If only random errors of the precision type are present in the results, the points may be expected to be distributed equally among the four quadrants because positive and negative deviations are equally likely. If systematic errors are present, the effect is to shift the points into the upper right and lower left quadrants. No matter where a point is located in the +− or −+ quadrants the addition (or subtraction) of a sufficiently large constant error will shift the point into a ++ (or −−) quadrant. The argument is then applied in reverse. If points are found predominantly in the ++ or −− quadrants, the presence of systematic errors is established.

It is instructive to consider results that are without systematic errors. The points would then be clustered compactly in a circular pattern centered on the intersection of the two lines. If systematic errors of varying magnitudes, both plus and minus, are now added to the results, the points move outward from the center more or less closely along a

45-deg line drawn through the intersection of the two lines. In fact, points will be off this 45-deg line solely because of the precision error that scattered them in the original compact circle.

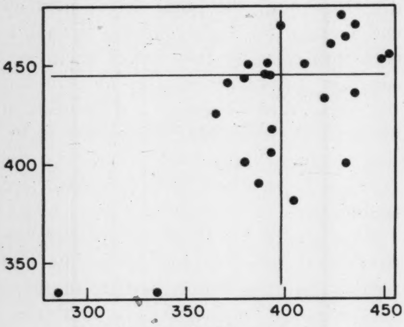


FIG. 1.—Tensile Strength, psi.

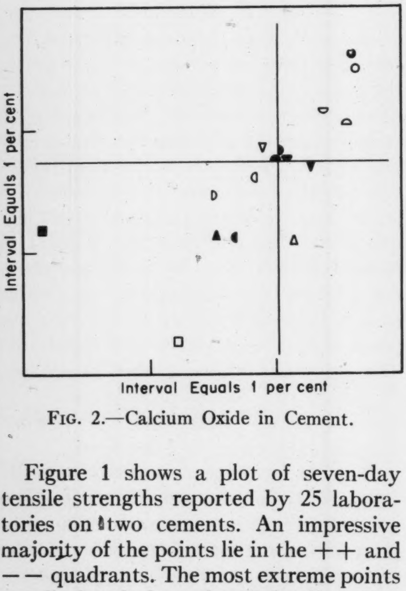


FIG. 2.—Calcium Oxide in Cement.

Figure 1 shows a plot of seven-day tensile strengths reported by 25 laboratories on two cements. An impressive majority of the points lie in the ++ and −− quadrants. The most extreme points are displaced along the 45-deg line. Certainly the two lower points provide a strong hint to two of the laboratories that they are troubled with large negative systematic errors. The possible contention of nonuniform material and sample troubles can be dealt with quickly. The materials must be satisfactorily uniform, otherwise there would exist high and low samples. High and low samples will be equally frequent. When the pairs of samples are sent out, each laboratory would receive one of the four sample combinations, ++, +−, −+, −−. These four combinations are all equally likely and all four quadrants would then be about equally populated with points. This equality of distribution is not present so that it is concluded that the material is reasonably uniform.

Figure 2 shows the results from eight laboratories reporting on calcium oxide in cement on two sets of paired samples. The solid symbols identify the laboratory results on the first pair of materials analysed. Two months later another pair of materials were analysed and the hollow symbols were plotted on another piece of graph paper using the appropriate range of values but the same unit interval for 1 per cent of calcium oxide. The two graphs were then superimposed so that the quadrant forming lines were coincident and all the points plotted on one graph. The configuration of the points, not the absolute values, is the important aspect of the plot. Notice how the points congregate in the ++ and −− quadrants. Clearly the circle laboratory and the square laboratory run true to form. The evidence is convincing that the circle laboratory gets results that are consistently higher than those obtained by other laboratories and the square laboratory gets low results. All this is established with just four results from each laboratory. The 45-deg line provides a reasonably good fit to the points so that some of the other laboratories undoubtedly have smaller systematic errors that are, in part, obscured by the random precision error.

Practically all tests gave patterns which showed a generally elliptical character with the long axis of the ellipse at 45-deg through the point plotted for the median (or average) values for the two materials. The degree of eccentricity of the ellipse is informative. The more elongated ellipses indicate that the test procedure is very vulnerable to individual interpretations or modifications that introduce systematic errors into the test procedure. A more careful specification of the test procedure or perhaps some change in the procedure may be needed to correct the situation.

THE IMPROVEMENT OF TEST RESULTS

The preceding section pointed out two ways in which progress may be made in reducing the scatter of the results reported by laboratories testing the same material. A relatively small amount of data, plotted as a joint scatter diagram for two materials, permitted the consideration of all three elements that have to be considered in seeking the causes for an undesirably large dispersion among the reported results. First, some assurance is needed that lack of uniformity in the material distributed is not mainly responsible for the scatter. This is a problem of some difficulty where the samples are perforce large and the laboratories numerous. Second, the identification of systematic errors as the cause for the more divergent results is easily made and the singling out of the responsible laboratories is a simple matter. Finally, faulty test procedures are likely to be the trouble when most of the points determined by the test results are strung along the 45-deg line in the graph. Improvement can only come by obtaining satisfactory evidence of the presence and causes of errors in test results and then taking whatever remedial steps are needed.

SCORING SYSTEM FOR TEST RESULTS

At the outset of the work a computation procedure was instituted to calculate the standard deviation for each test for each cement. The 103 laboratories available provided an unusually large basis for calculating the standard deviation of the hundred or so results as reported. Only the most obvious blunders were deleted before making the computation. It was realized that a more careful screening of the data was desirable, but there was considerable hesitation about deleting results and thereby obtaining an estimate of the standard deviation that might be too small. In the scoring scheme about to be described the above standard deviation was used with the idea of determining whether the distribution of the test results could be satisfactorily described using this estimate of the standard deviation. The scoring scheme also greatly reduced the amount of arithmetic required in dealing with over 24,000 test results. The scores made for simplicity in comparing tests because the individual units used in reporting results were replaced by multiples of the appropriate standard deviation.

TABLE II.—SHOWS HOW (1) SCORE DEPENDS ON DEVIATION FROM THE AVERAGE AND (2) THE PROBABILITY OF ACHIEVING THE SCORE.

Difference Between Result and Average	Score	Probability
0–1.0 σ	4	0.69
1.0 σ–1.5 σ	3	0.18
1.5 σ–2.0 σ	2	0.09
2.0 σ–2.5 σ	1	0.03
over 2.5 σ	0	0.01

A test result within one standard deviation of the average was given a score of 4—plus if high, minus if low. Results more than one standard deviation away but within one and a half standard deviations were given a score of 3. The complete scoring system is shown in Table II.

The appended column of probabilities gives the chances that a laboratory will receive any one of these five scores if the laboratory has in fact a standard deviation equal to that computed from all the data. One laboratory in a hundred may deviate by 2.5 σ, and receive a score of zero, even if its work is up to par. A single set of scores is not very informative. If as many as ten scores have been awarded, then the average score becomes meaningful. A laboratory might have an average of 4.0 for the ten physical (or chemical) tests on one cement but only if it scored 4 on every test. The chance of this happening is the tenth power of 0.69 or 0.0245. One in forty laboratories will amass such an impressive average, even though its work is no better than the standard deviation used in setting up the scoring. The numerical work is tedious but the chances of getting various average scores based on ten tests can be computed. These chances are displayed in Table III, column 2, which gives the expected number of laboratories per hundred making each average score.

TABLE III.—EXPECTED NUMBER OF LABORATORIES PER HUNDRED MAKING INDICATED AVERAGE SCORES BASED ON TEN TEST RESULTS.

Average Score	Number of Laboratories		
	Computed σ	80 per cent of Computed σ	Adjusted to 94 Laboratories
4.0	2.45	9.35	8.78
3.9	6.38	17.77	16.00
3.8	10.68	20.89	19.64
3.7	13.77	18.74	17.62
3.6	14.92	14.00	13.16
3.5	14.10	9.05	8.52
3.4	11.93	5.22	4.91
3.3	9.18	2.72	2.56
3.2	6.51	1.30	1.22
3.1	4.28	0.58	0.55
3.0	2.55	0.23	0.22
2.9	1.52	...	...
2.8	0.82	...	...
2.7	0.43	...	...
2.6	0.21	...	...
Total	99.73	99.85	93.88

The computed frequencies, listed in column 2, Table III are represented graphically by the outline bars in Fig. 3. When this theoretical curve was compared with the observed distribution of the scores made by the 100 laboratories, pronounced discrepancies were found. Invariably the theoretical curve underestimated the number of high scores. Also, in theory, in studies of this size, no scores were expected below 2.6. In fact, several such scores were usually found. Computations showed that the deletion of six low-scoring laboratories would reduce the standard deviation by at least 20 per cent. If the smaller standard deviation is used, the chances of achieving various scores on a single test changes from those given in the last column of Table II to the following:

Score	Probability
4	0.789
3	0.150
2	0.048
1	0.011
0	0.002

The chance of a laboratory achieving an average of 4.0 is now the tenth power of 0.789 or 0.0935. The number of laboratories out of 94 that can be expected to score 4.0 is 94 × 0.0935 or 8.78 laboratories. The expected number of laboratories obtaining various scores using this smaller standard deviation are shown in the last column of Table III. The expected number of laboratories making these scores are shown by the solid bars in Fig. 3. The contrast in the two distributions is pronounced especially in the sharp increase in the expected number of laboratories making high scores. The solid bar chart is a much better approximation to the observed distribution of laboratory average scores and is confirmed by the high proportion in the scoring. All in all the evidence is unmistakable that about 90 per cent of the laboratories are performing the test in such a way that the over all scatter of their results is associated with a standard deviation about 20 per cent below that calculated from all the laboratories. (All the laboratories here means after discarding two or three patently absurd results arising from what appear to be typing or computing slips.)

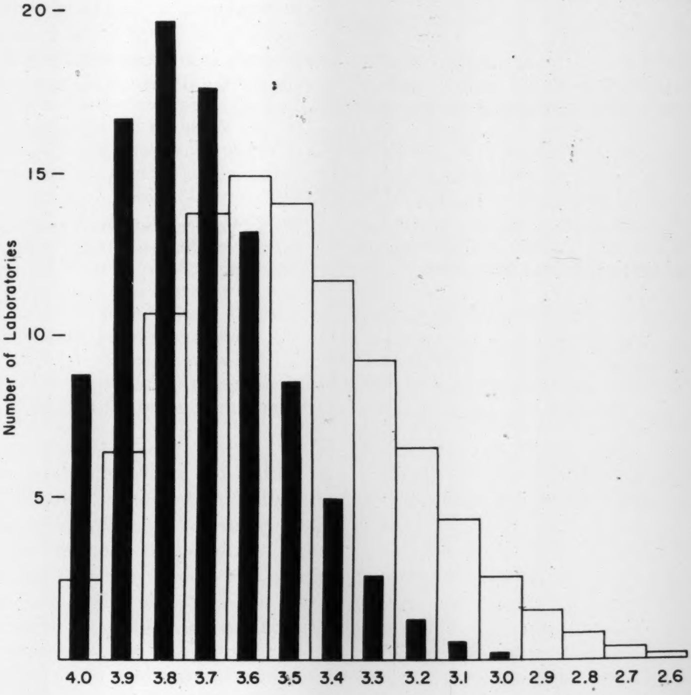


FIG. 3.—Average Score for Ten Results.

The distribution of the averages of *ten* scores was chosen because initially averages were taken of the ten physical (or ten chemical) scores by a laboratory on one cement. There were twelve cement samples but sometimes a pair was skipped. The theoretical distribution for averages based on twelve results differs very slightly from that given for averages of ten.

The standard deviation is a property of the test procedure, provided the test is performed with due care with the specified equipment. A few laboratories by lack of care or by unwitting departures from the specified method of performing the test can obtain results sufficiently out of line to inflate substantially the estimated standard deviation. The present study has established beyond any possibility of controversy that a handful of laboratories is responsible for making the test procedures appear substantially less satisfactory than they really are.

SUMMARY

A graphic procedure using paired samples has been developed to demonstrate to what extent systematic errors are present in interlaboratory tests. This procedure also indicates the extent of random errors and the ultimate precision of a test procedure.

A scoring system for evaluating the test results of a group of laboratories has been developed. It has been shown that the results of a few laboratories doing poor work can substantially inflate the apparent standard deviation obtained for any test.

REFERENCES

(1) J. R. Crandall and R. L. Blaine, "Statistical Evaluation of Interlaboratory Cement Tests," see p. 1129, this publication.

(2) W. J. Youden, *Industrial and Engineering Chem.*, Vol. 50, p. 63a, Aug.; p. 91a, Oct.; p. 77a, Dec. 1958.

STATISTICAL EVALUATION OF INTERLABORATORY CEMENT TESTS*

By J. R. Crandall[1] and R. L. Blaine[1]

Synopsis

A cooperative series of physical and chemical tests was made by 103 laboratories on 12 samples of cement during the period of 1 yr. Results of tests by all laboratories on each property were plotted in scatter-diagrams according to the Youden method to enable the participating laboratories to evaluate their results quickly. The results were treated statistically. Both statistical and graphic methods were employed to indicate the precision of the test methods and the extent of laboratory bias. The lack of precision with some of the tests indicated a need for improved test methods.

The work of each of the various laboratories was evaluated by a rating system. A large number of laboratories obtained results that were in good agreement, but a few laboratories showed poor agreement with the larger group. Many of the discrepant laboratories did not improve during the 1-yr period the test program was active.

Discrepant results obtained by a few laboratories inflated appreciably the standard deviations among laboratories for the various tests.

The need for a continuing reference sample program was indicated.

The values obtained in the testing of cement, as those obtained in testing of most materials, are subject to many variables. The methods of performing the tests are carefully specified as well as the requirements and tolerances for test equipment to be used. Cement testing laboratories have, in addition, had a unique service for the past 30 years in that the Cement Reference Laboratory (CRL) has inspected the equipment and instructed the personnel of these laboratories at regular intervals. A number of comparative samples have also been distributed by this group during the past 30 years. A report of the work done by the CRL and of the resulting improvements in test equipment was presented by J. R. Dise.[2]

The service of the CRL of inspecting laboratory equipment, instructing personnel and demonstrating test methods is costly; furthermore, because turnovers occur in testing personnel and changes occur in test methods and equipment, more frequent inspections would be desirable.

The CRL comparative tests as well as other interlaboratory tests have indicated that there are still many and large discrepancies between the results as reported by different laboratories. It has not been clear from previous comparative tests if these discrepancies resulted from normal variations to be expected or if laboratory bias was involved to any appreciable extent.

---

* Presented at a special session on Statistical Treatment of Interlaboratory Test Results, Including a New Graphical Method sponsored by Committee C-1 on Cement held on June 25, 1959 during the Sixty-second Annual Meeting of the Society.

[1] Concreting Materials Section, National Bureau of Standards, Washington, D. C.

[2] J. R. Dise, "Cement Reference Laboratory (1929–1959)," see p. 369, this publication.

In order to determine the reproducibility of the various tests for cement now in use and to evaluate the nature of the discrepancies, as well as to offer a means for supplementing the work of the CRL, a Cement Reference Sample program was proposed at the 1957 ASTM Annual Meeting, to the Subcommittee on the CRL of Committee C-1, by the personnel of the National Bureau of Standards (NBS).

The Reference Sample Program was considered an extension of the NBS Standard Sample Program which has served laboratories everywhere for almost 60 yr. Before these standard samples of cement could be issued, however, it was necessary to solve problems relative to blending, packaging and distribution of the samples as well as tabulation, evaluation and presentation of the test results. Although the planned program of interlaboratory tests was quite extensive, it was necessary to limit the number of participating laboratories because of the present limited facilities for blending very large quantities of materials.

TABLE I.—MANUFACTURERS OF CEMENTS USED.

Universal Atlas Cement Co.
North American Cement Corp.
Standard Lime & Cement Co.
Lehigh Portland Cement Co.
Alpha Portland Cement Co.
Green Bay Division, Pittsburgh Coke & Chemical Co.
Lone Star Cement Co.
Medusa Portland Cement Co.

PARTICIPATING LABORATORIES

It was desirable in a program of this kind to have as broad a distribution of laboratories as possible, both with respect to interest and geography. Included in the program were both research and mill-control laboratories of cement manufacturers, State Highway laboratories, Federal laboratories, and commercial laboratories. Included among the cement manufacturer laboratories were larger companies who regularly conduct interlaboratory tests and smaller companies who have only one or two mills and whose opportunities for interlaboratory tests ordinarily are limited. All participating laboratories had been inspected by the CRL. There were 57 cement manufacturer laboratories, 24 State Highway laboratories, 11 commercial laboratories and 11 Federal Government laboratories, making a total of 103 laboratories from all sections of the country. A list of the participating laboratories is given in Appendix I.

TABLE II.—IDENTIFICATION OF PAIRS OF CEMENT SAMPLES.

Sample	Type	Lot Number	Date Shipped to Laboratories
No. 1......	I	1	March 1958
No. 2......	I	2	
No. 3......	I	3	May 1958
No. 4......	IA	4	
No. 5......	II	5	July 1958
No. 6......	IS	6	
No. 7......	III	7	Sept. 1958
No. 8......	IA	8	
No. 9......	II	9	Nov. 1958
No. 10.....	V	10	
No. 11.....	IA	4	Jan. 1959
No. 12.....	I	3	

TEST PROGRAM

The test program consisted of physical and chemical tests by the 103 laboratories on 12 cement samples. Ten lots of different cements were used to prepare the 12 samples.

Nine of the ten cements used in the program were purchased from suppliers in the Washington, D. C., area, and one cement was donated by the manufacturer (Table I). Each lot of cement was from a single carload shipment. Six types of cement were used (Table II). Each lot consisted of 16 or 18 bags of cement, except lots 3 and 4, which were double that amount. Each cement was sieved through a No. 20 vibrating screen and each lot then blended for 2 to 3 hr in a 20-cu ft Patterson-Kelly blender equipped with an intensifier.

The cement was then packaged in plastic-lined, canvas bags, approximately 12 lb per package, and stored in sealed steel drums until shipment, except in the case of samples Nos. 11 and 12, where one half of each set of samples was stored on a skid in a storeroom. Tests for uniformity were made on every tenth sample bag packaged. Four separate test samples were taken from each of these bags, two being used for air permeability fineness tests and the other two for SO_3 determinations. Each sample was coded so that the test operator did not know which of the samples were duplicates from the same bag.

Uniformity tests were made comparing the differences between duplicate determinations on the same samples, the sums of duplicates, and the successive differences between sums. All these tests for uniformity were made in the NBS Washington laboratory, except tests on one pair of samples which were made in the NBS Seattle laboratory. In each instance, the statistical analysis indicated that the cement was well-blended.

TABLE III.—TEST METHODS USED.

Normal consistency of hydraulic cement	ASTM Method C 187 - 58[b]
Soundness, autoclave	ASTM Method C 151 - 58[b]
Time of setting, Gillmore	ASTM Method C 266 - 58 T[b]
Air content	ASTM Method C 185 - 58 T[b]
Compressive strength (6-cube batch)	ASTM Method C 109 - 58[b]
Fineness, air permeability	ASTM Method C 204 - 55[b]
Tensile strength[a]	ASTM Method C 190 - 58[b]
Fineness turbidimeter[a]	ASTM Method C 115 - 58[b]
Heat of hydration of portland cement[a]	ASTM Method C 186 - 55[b]
Time of setting, Vicat needle[a]	ASTM Method C 191 - 58[b]
False set[a]	ASTM Method C 359 - 56 T[b]
Sulfate resistance expansion[a]	Experimental Test

Test Procedure	ASTM Methods	Section of the Method Used for Samples Nos. 1 to 8	Section of the Method Used for Samples Nos. 9 to 12
Silicon dioxide (SiO_2)	C 114 - 58[b]	33	8
Aluminum oxide (Al_2O_3)	C 114 - 58[b]	12	12
Ferric oxide (Fe_2O_3)	C 114 - 58[b]	10 and 11	10 and 11
Calcium oxide (CaO)	C 114 - 58[b]	34 and 35	13
Magnesium oxide (MgO)	C 114 - 58[b]	36	14 and 15
Sulfur trioxide (SO_3)	C 114 - 58[b]	16	16
Loss on ignition	C 114 - 58[b]	20	20
Insoluble residue	C 114 - 58[b]	28	28
Sodium oxide (Na_2O)	C 114 - 58 T[b]	15 through 18	[c]
Potassium oxide (K_2O)	C 114 - 58 T[b]	15 through 18	[c]
Manganic oxide (Mn_2O_3)[a]	C 114 - 58[b]	49 and 50	...
Sulfide sulfur[a]	C 114 - 58[b]	17 through 19	...

[a] These properties determined on one pair of samples only.
[b] 1958 Book of ASTM Standards, Part 4.
[c] A Proposed Interim Federal Specification for Cement.

ASTM test methods were used throughout the program, except as indicated in Table III. For samples Nos. 9 to 12, ASTM referee test methods were used for the analytical determinations, with Federal Specifications for the alkalies. In each case, the laboratories were requested to report results of single determinations.

Samples of two of the cements were supplied to the participating laboratories at 2-month intervals for a period of 1 yr.

TABLE IV.—STANDARD DEVIATIONS AND COEFFICIENTS OF VARIATION FOR DATA REPORTED BY ALL LABORATORIES.

Sample	No. 1		No. 2		No. 3		No. 4		No. 5		No. 6	
	S.D.	C.V.	S.D.	C.V.	S.D.	C.V.	S.D.	C.V.	S.D.	C.V.	S.D.	C.V.
Water consistency, per cent	0.45	1.8	0.86	3.4	0.50	1.9	0.58	2.2	0.71	2.9	1.2	4.5
Initial setting, Gillmore, hr	0.59	18	0.64	18	0.40	14	0.47	14	...	...	...	...
Final setting, Gillmore, hr	0.92	17	0.89	16	0.80	16	0.85	16	...	...	...	...
Expansion, per cent	0.04	26	0.03	23	0.04	55	0.03	34	0.008	21	0.01	38
Air entrainment, per cent	1.4	20	1.3	15	1.1	14	1.9	12	1.2	12	1.4	12
Water-air entrainment, per cent	3.6	5.1	3.4	4.9	9.5	5.1	4.9	8.1	2.6	3.8	3.3	4.9
Compressive strength, 3 days, psi	221	9.5	276	9.7	250	9.0	237	11	163	8.6	231	10
Compressive strength, 7 days, psi	305	8.1	234	6.4	304	7.2	280	7.8	206	7.7	313	8.6
Water compressive strength, per cent	1.4	2.8	1.3	2.7	1.2	2.5	1.2	2.5	1.3	2.8	1.5	3.1
Fineness, *B*, sq cm per g	105	3.2	90	2.6	177	4.6	107	3.5	112	3.2	304	6.7
SiO_2, per cent	0.16	0.76	0.21	0.95	0.18	0.84	0.20	0.91	0.19	0.91	0.24	1.1
Al_2O_3, per cent	0.36	5.3	0.33	6.8	0.97	6.2	0.29	4.9	0.35	6.5	0.45	5.6
Fe_2O_3, per cent	0.20	8.1	0.24	8.3	0.22	7.9	0.21	7.5	0.12	2.5	0.10	4.1
CaO, per cent	0.48	0.76	0.43	0.69	0.39	0.61	0.43	0.66	0.87	1.4	0.70	1.3
MgO, per cent	0.21	7.3	0.28	7.4	0.15	8.3	0.14	10	0.25	11	0.24	6.2
SO_3, per cent	0.11	6.3	0.12	4.3	0.10	5.3	0.09	5.2	0.14	7.6	0.13	5.6
Ignition loss, per cent	0.12	16	0.10	13	0.14	12	0.16	21	0.22	16	0.37	14
Residue, per cent	0.15	94	0.08	32	0.10	65	0.17	96	0.08	45	0.10	20
Na_2O, per cent	0.03	8.7	0.03	11	0.03	15	0.03	19	0.03	14	0.03	36
K_2O, per cent	0.04	5.1	0.07	7.5	0.05	6.4	0.04	5.1	0.04	8.2	0.03	15
Mn_2O_3, per cent	...	...	...	...	...	...	...	...	0.07	82	0.09	15
Sulfide sulfur, per cent	...	...	...	...	...	...	...	...	0.01	71	0.07	12
False set, initial mm	...	...	...	...	...	...	...	...	...	...	...	...
False set, final mm	...	...	...	...	...	...	...	...	...	...	...	...
Tensile strength, 3 days, psi	...	...	...	...	32	9.1	29	9.1	...	...	...	...
Tensile strength, 7 days, psi	...	...	...	...	36	8.6	29	7.4	...	...	...	...
Water tensile strength, per cent	...	...	...	...	0.08	0.72	0.19	1.8	...	...	...	...
Vicat setting, hr	...	...	...	...	...	...	...	...	0.37	14	71	24
Fineness, Wagner, sq cm per g	...	...	...	...	...	...	...	...	...	...	...	...
Retained on No. 325 sieve, g	...	...	...	...	...	...	...	...	...	...	...	...
Sulfate resistance expansion, 14 days, per cent	...	...	...	...	...	...	...	...	...	...	...	...
Sulfate resistance expansion, 28 days, per cent	...	...	...	...	...	...	...	...	...	...	...	...
Heat of hydration, 7 days cal per g	...	...	...	...	...	...	...	...	...	...	...	...
Heat of hydration, 28 days, cal per g	...	...	...	...	...	...	...	...	...	...	...	...

S.D. Standard deviation.
C.V. Coefficient of variation, per cent.

Sample	No. 7		No. 8		No. 9		No. 10		No. 11		No. 12	
	S.D.	C.V.	S.D.	C.V.	S.D.	C.V.	S.D.	C.V.	S.D.	C.V.	S.D.	C.V.
Water consistency, per cent	1.1	3.9	0.59	2.4	0.44	1.8	0.94	3.7	0.58	2.3	0.58	2.2
Initial setting, Gillmore, hr	0.39	14	0.44	15	0.51	14	0.85	18	0.46	14	0.45	15
Final setting, Gillmore, hr	0.81	17	0.87	17	0.82	14	1.3	17	0.69	13	0.64	13
Expansion, per cent	0.03	115	0.04	23	0.01	24	0.01	30	0.12	132	0.05	87
Air entrainment, per cent	1.4	20	1.2	6.2	1.3	12	1.4	15	1.5	9.1	1.3	13
Water-air entrainment, per cent	2.5	3.6	2.9	5.0	2.9	4.3	3.0	4.4	2.8	4.8	2.1	3.0
Compressive strength, 3 days, psi	240	9.0	179	10	156	8.4	193	8.4	209	9.1	254	9.2
Compressive strength, 7 days, psi	333	6.9	294	12	248	8.9	293	8.7	293	7.9	320	7.4
Water, compressive strength, per cent	1.2	2.5	1.3	2.9	1.3	2.8	1.4	3.0	1.1	2.4	1.1	2.3
Fineness, *B*, sq cm per g	279	6.1	90	2.9	121	3.6	123	4.2	135	4.3	109	2.8
SiO_2, per cent	0.21	1.0	0.22	0.99	0.36	1.7	0.34	1.5	0.34	1.6	0.25	1.2
Al_2O_3, per cent	0.20	3.6	0.30	5.9	0.29	5.1	0.36	9.7	0.22	3.7	0.24	4.0
Fe_2O_3, per cent	0.09	3.6	0.08	3.3	0.11	2.5	0.15	3.5	0.10	3.7	0.09	3.4
CaO, per cent	0.34	0.52	0.32	0.50	0.42	0.67	0.42	0.64	0.45	0.69	0.41	0.64
MgO, per cent	0.15	7.8	0.12	3.4	0.27	23	0.20	26	0.18	13	0.21	11
SO_3, per cent	0.13	4.4	0.11	5.1	0.09	4.5	0.09	5.3	0.06	3.4	0.08	4.5
Ignition loss, per cent	0.15	12	0.10	12	0.14	13	0.10	14	0.11	13	0.14	11
Residue, per cent	0.06	30	0.06	37	0.08	55	0.07	54	0.06	46	0.05	40
Na_2O, per cent	0.04	30	0.04	13	0.03	20	0.07	13	0.03	19	0.03	16
K_2O, per cent	0.04	5.2	0.08	7.5	0.04	4.7	0.05	24	0.05	5.8	0.04	5.8
Mn_2O_3, per cent	...	...	...	...	...	...	...	...	...	...	...	...
Sulfide sulfur, per cent	...	...	...	...	...	...	...	...	...	...	...	...
False set, initial mm	...	...	...	...	...	...	...	...	5.6	18	6.6	20
False set, final mm	...	...	...	...	...	...	...	...	5.4	27	9.0	48
Tensile strength, 3 days, psi	...	...	...	...	...	...	...	...	...	...	...	...
Tensile strength, 7 days, psi	...	...	...	...	...	...	...	...	...	...	...	...
Water tensile strength, per cent	...	...	...	...	...	...	...	...	...	...	...	...
Vicat setting, hr	...	...	...	...	...	...	...	...	...	...	...	...
Fineness, Wagner, sq cm per g	119	4.7	95	5.5	...	...	...	...	...	...	...	...
Retained on No. 325 sieve, g	0.02	131	0.02	18	...	...	...	...	...	...	...	...
Sulfate resistance expansion, 14 days, per cent	...	...	...	...	0.002	38	0.001	49	...	...	...	...
Sulfate resistance expansion, 28 days, per cent	...	...	...	...	0.022	123	0.011	122	...	...	...	...
Heat of hydration, 7 days, cal per g	...	...	...	...	4.2	6.1	5.1	7.9	...	...	...	...
Heat of hydration, 28 days, cal per g	...	...	...	...	7.8	9.5	8.9	11	...	...	...	...

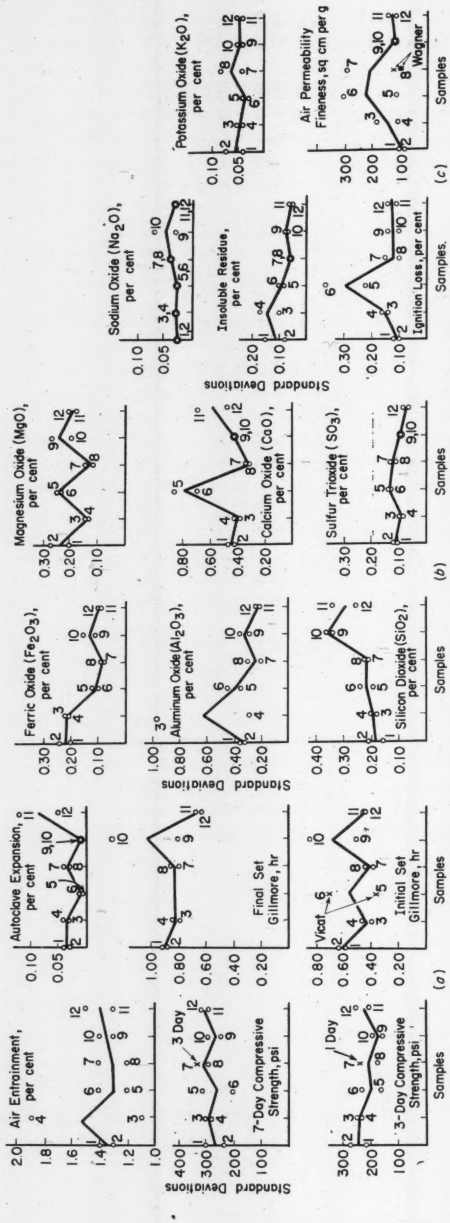


FIG. 1.—Average Standard Deviations for the Six Successive Pairs of Cement Samples.

The participating laboratories were requested to make the physical and chemical tests as required by detailed instructions furnished with each pair of samples and to report the results to the National Bureau of Standards within 1 month after receiving the samples. With the instructions for the first pair of samples it was also requested that the same operator and the same analyst of each laboratory also make all subsequent test determinations.

As the reports were received and the tabulated results scanned, those laboratories whose result appeared to contain an error were asked to confirm the questionable value. In most instances, suspicion of the presence of an error was confirmed in time to permit inclusion of the corrected value in the program. At the NBS, the results were tabulated, the data placed on IBM cards, and the averages, standard deviations between laboratories, and coefficients of variation computed by a digital computer. Scatter diagrams as proposed by Youden[3] were plotted for each set of test results. The scatter diagrams, averages, standard deviations, and coefficients of variation were furnished each of the participating laboratories at the time the next pair of samples was distributed. This afforded the personnel of the participating laboratories an opportunity to study the results of the previous tests and make procedural corrections before making tests on the new samples.

RESULTS OF TESTS

The standard deviations between laboratories and corresponding coefficients of variation for data reported by all laboratories are presented in Table IV. The values for standard deviation are also presented in the graphs shown in Figs. 1(*a*), (*b*), and (*c*). It may be noted that considerable differences exist in the variability among laboratories for the different tests, for the different samples in pairs, and for the different pairs. For most tests there was no definite evidence of improvement of results as the program proceeded. The use of referee chemical methods did not result in better concordance than was obtained with the use of optional chemical methods. The average values for the various tests on the different samples are presented in Appendix II.

Scatter diagrams were prepared in accordance with the method proposed by Youden[3] in which the value for a property of one sample is plotted on the horizontal axis and the corresponding value for the other sample on the vertical axis. Thus the results for one property for two samples from each laboratory are represented by a point on the diagram. Each diagram is divided into quadrants by a vertical and a horizontal line to indicate the medians. That is, there are as many laboratories to the left of the vertical line as to the right and there are as many laboratories above the horizontal line as below.

Figure 2 shows the distribution of results for the percentage loss on ignition of cement samples Nos. 11 and 12. The pattern formed by the points is fairly circular and the crossed center lines are the median values for the samples. Only two of the laboratories had ignition loss results for these two cements that deviated considerably from the median values, and there was a fairly uniform distribution of the points. Under ideal circumstances, there should be nearly an equal number of points in each of the four quadrants and the pattern of points should be roughly circular, as shown in Fig. 2.

---

[3] W. J. Youden, "A New Graphic Method for Statistical Treatment and Evaluation of Interlaboratory Tests," "Application of the Graphic Method to Other Interlaboratory Testing and Specifications." These papers presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959, have been combined under the title "Statistical Aspects of the Cement Testing Program," see p. 1120, this publication.

When this method of presentation is used, it is an easy matter for a participating laboratory to determine the relationship of its results to the median values and to the results obtained by the other laboratories.

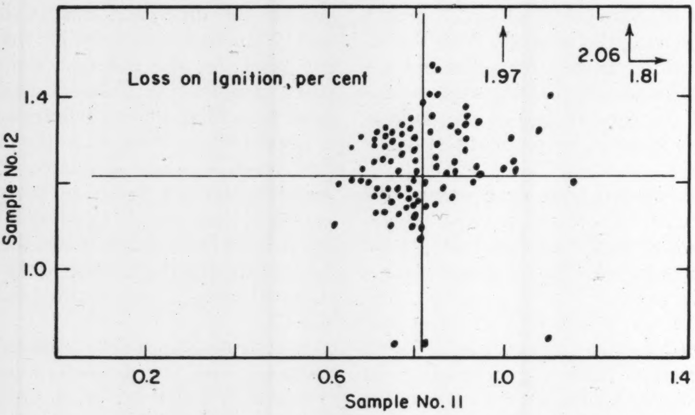


FIG. 2.—Scatter Diagram for Percentage Loss on Ignition for Samples Nos. 11 and 12.

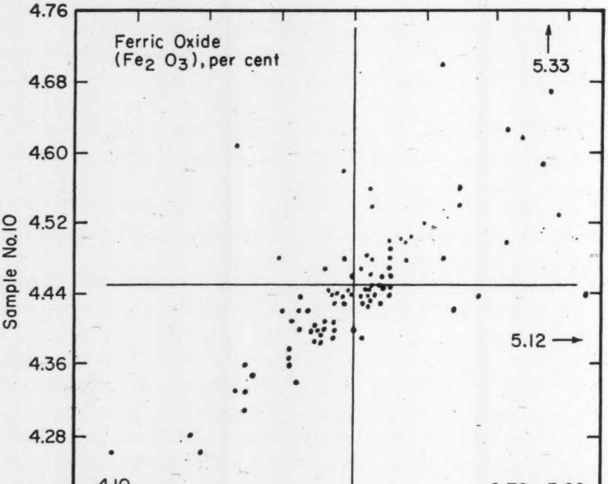


FIG. 3.—Scatter Diagram for Percentage Ferric Oxide (Fe_2O_3) for Samples Nos. 9 and 10.

If the scatter of points is oval, as indicated in Fig. 3, with the long axis of the oval at approximately 45 deg, passing through the lower left quadrant and the upper right quadrant, there is evidence of a strong correlation between the test results obtained in any one laboratory. The majority of the scatter diagrams tended to be oval along the 45 deg axis. This indicates that many of the laboratories have a tendency to obtain high values on both samples if high val-

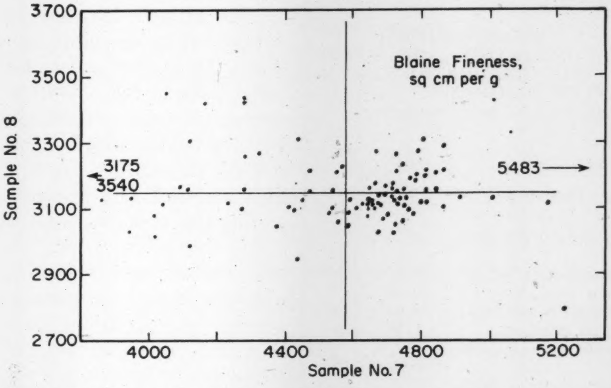


FIG. 4.—Scatter Diagram for Air-Permeability Fineness for Samples Nos. 7 and 8.

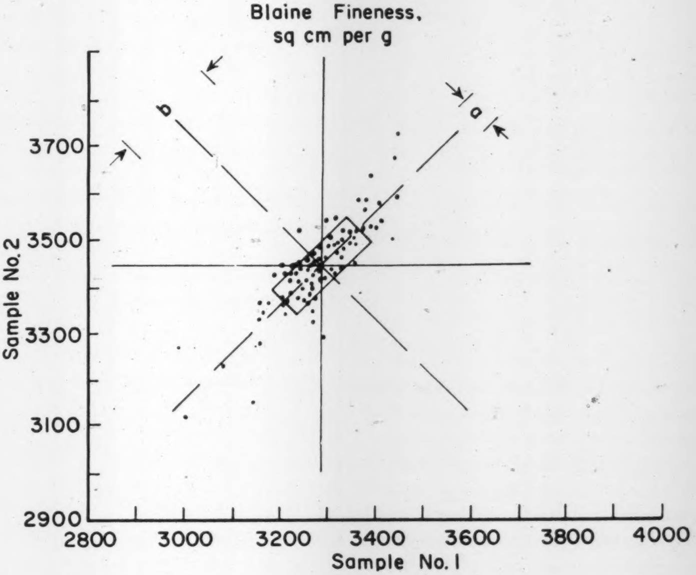


FIG. 5.—Scatter Diagram for Air-Permeability Fineness for Samples Nos. 1 and 2. The significance of parameters "*a*" and "*b*" is explained in the text.

ues are obtained on one. In this particular case, there should be 25 points in each quadrant. However, the lower left and upper right quadrants together account for about 80 points, which is evidence of numerous laboratory biases. Among the possible sources of such errors may be the ambiguity or misinterpretation of the test methods. With a chemical analysis such as this, errors in standardization of solutions or possibly constant errors in weighing could contribute to the bias. With uniform samples, a broader oval normally indicates a greater imprecision on the part of the operator or apparatus. Laboratories whose results are consistently represented by points fairly far out in the upper right or lower left quadrant are believed to have a systematic error in the test procedure or apparatus.

TABLE V.—MEASUREMENT OF STANDARD DEVIATION OF TEST METHOD AND LABORATORY BIAS FROM SCATTER DIAGRAMS.

Averages for All Twelve Samples

	a	*b/a*
Physical Tests		
Normal consistency, per cent of H_2O	1.0	1.47
Time of setting, initial, hr	0.72	2.03
Time of setting, final, hr	0.80	2.71
Autoclave expansion, per cent	0.023	1.56
Air entrainment, per cent	1.26	2.50
Compressive strength, 3 days, psi	255	1.86
Compressive strength, 7 days, psi	314	2.12
Fineness, sq cm per g	162	1.85
Chemical Tests		
Silicon dioxide (SiO_2), per cent	0.17	2.17
Aluminum oxide (Al_2O_3), per cent	0.26	2.24
Ferric oxide (Fe_2O_3), per cent	0.07	2.33
Calcium oxide (CaO), per cent	0.25	2.83
Magnesium oxide (MgO), per cent	0.14	2.14
Sulfur trioxide (SO_3), per cent	0.07	1.97
Ignition loss, per cent	0.18	1.58
Insoluble residue, per cent	0.06	2.35
Sodium oxide (Na_2O), per cent	0.02	2.96
Potassium oxide (K_2O), per cent	0.05	2.10

Figure 4 shows a pattern which indicates no laboratory bias, but a greater spread for one sample than for the other. This results in a pattern which is oval in form with the long axis either vertical or horizontal, depending on which sample shows the greater scatter for the test results. Sample No. 7 was a high early-strength cement, and it is quite apparent that the laboratories had difficulty determining the surface areas of this much finer material. Few instances were found having this type of scatter diagram.

An evaluation of both the laboratory bias and the test method precision was obtained from the parameters of rectangles constructed as shown in Fig. 5. The sides of the rectangles were formed by pairs of lines drawn parallel to and equidistant from the intersecting 45 deg lines. For each direction, the parallel lines were spaced so that approximately 68 per cent of the points fell between them. This 68 per cent should be within plus or minus one standard deviation in a normal distribution. The magnitude of a is an indication of the imprecision of the test. A ratio of b/a greater than 1 is an indication of a laboratory bias which is significantly greater than the random error of the test. Obviously, it would be desirable to have the ratio b/a as close to 1 as possible, and it would be desirable to have a as small as possible. This type of graph indicates where to look for possible improvement of test results.

The values for a and b/a obtained in this program are presented in Table V. The values are averages for all 12 samples. Tests for final time of set and air entrainment have the largest b/a ratio or laboratory bias in the physical tests, whereas Na_2O and CaO determinations have the largest b/a ratio in the chemical tests. The chemical tests had an average *b*/*a* ratio of 2.27, as compared to an average of 2.01 for the physical tests. A lack of precision in the test method itself, that is, a large value for *a*, would result in a lower *b*/*a* ratio.

After the tests on the first three pairs of samples had been completed, the average standard deviation for each of the tests was determined. Figure 6 is the scatter diagram for air entrainment for samples Nos. 7 and 8 with squares drawn using the standard deviation thus determined. The sides of the dotted inner square are plus and minus one times this standard deviation from the center. The sides of the outer square are at 1.95 times the standard deviation from the center, and this larger square should contain 90 per cent of all the points. These scatter diagrams were distributed to all the laboratories, giving them an opportunity to evaluate their own work in relation to that done by all the laboratories and to re-examine and improve their own procedures where need for improvement was indicated.

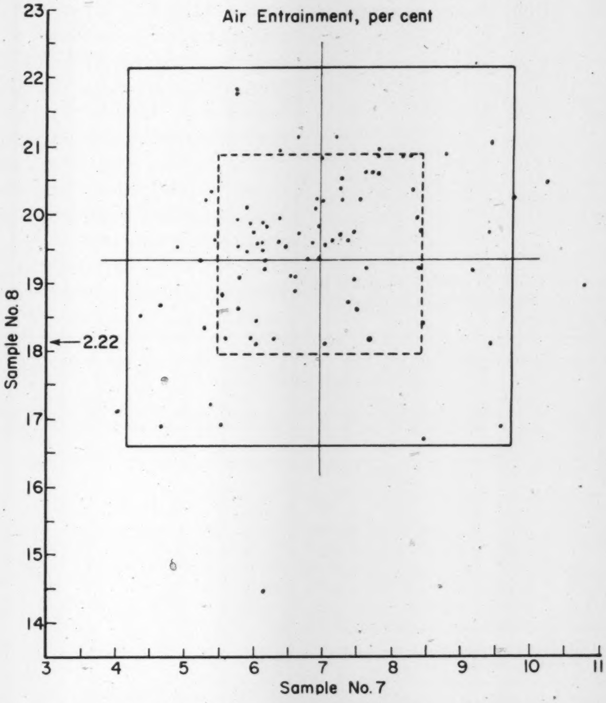


FIG. 6.—Scatter Diagram for Percentage Air Entrained in the 1:4 Mortars Reported for Samples Nos. 7 and 8.

The significance of the squares is explained in the text.

The graphic methods just discussed would enable a laboratory to determine its proficiency with respect to any one of the tests. In order to develop an evaluation of a laboratory's performance on all tests, the laboratories were rated on the basis of the averages and standard deviations obtained. A schematic diagram of the method used for rating the values reported by the various laboratories is shown in Fig. 7. The center block represents values within plus and minus one standard deviation of the mean. These values were assigned a rating of 4. Deviations from the mean of 1.0 to 1.5 times the standard deviations were assigned the rating 3. Between 1.5 and 2.0 standard deviations, the rating was 2, and from 2.0 to 2.5 standard deviations it was 1. Any value greater than 2.5 times the standard deviation was rated 0. Values to the right of center or above average were designated plus, and those to the left of center or below average were designated minus.

Table VI demonstrates the application of this rating system. Laboratory A, in determining normal consistency of neat pastes, and laboratory B, in determining SO_3 obtained values close to the averages of all laboratories for the tests in question. On the other hand, the results obtained by laboratory C, in determining normal consistency of neat pastes, indicates the presence of a consistent bias with respect to the average of all laboratories. Laboratory D, in determining autoclave expansion, had a very low rating at the start, but rapidly moved closer to the laboratory average. This laboratory had a tendency to obtain low results for this test. The ratings for laboratory E, determining 7-day compressive strength, show a great deal of variability. Laboratory F, determining normal consistency of neat pastes, was slightly below the general average.

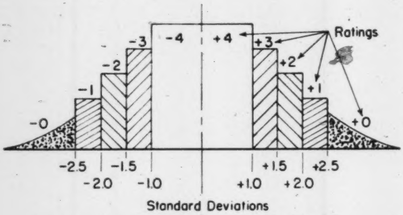


FIG. 7.—System Used to Assign Ratings to Laboratories on the Basis of Values Reported by All the Participants.

TABLE VI.—EXAMPLES OF RATINGS OBTAINED BY SIX OF THE LABORATORIES ON 12 SAMPLES.

Samples......		No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	No. 11	No. 12	Average[c]
Laboratory	Test													
A....	*a*[a]	+4	−4	+4	+4	+4	+4	−4	+4	+4	−4	−4	−4	4.00
B.....	*b*	4[b]	+4	+4	+4	+4	+4	−4	−4	4[b]	+4	−4	−4	4.00
C.....	*a*	+1	+3	+3	+0	+0	+2	+3	+0	+3	+2	+0	+0	1.42
D....	*c*	−0	−0	−4	−4	−3	4[b]	−4	4[b]	−3	+1	−4	−4	2.91
E.....	*d*	−4	+4	−1	−2	−4	−0	+2	+4	+2	+1	−3	−2	2.42
F.....	*a*	+2	+2	−4	−3	...	...	−3	+4	−4	−4	−1	+3	3.00

[a] Tests *a* = Normal consistency.
b = Sulfur trioxide (SO_3).
c = Autoclave expansion.
d = Compressive strength, 7 days.

[b] Identical with average for the test for this sample.

[c] Average computed without regard to signs.

These data indicate the value of a continuous reference sample program in helping a laboratory to continually evaluate its results.

Average values for ratings (without regard to signs) were computed for all the physical and all the chemical tests for each laboratory for each sample tested.

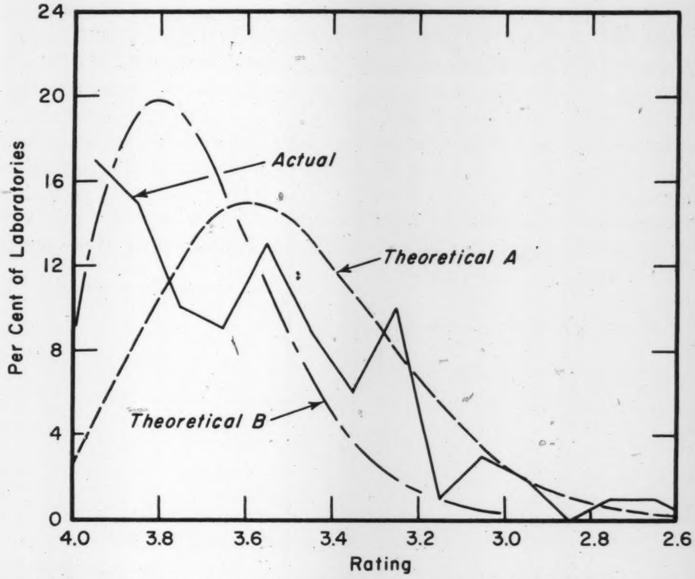


FIG. 8.—Frequency Distribution of Average Laboratory Ratings for All Physical Tests Reported for Sample No. 6.

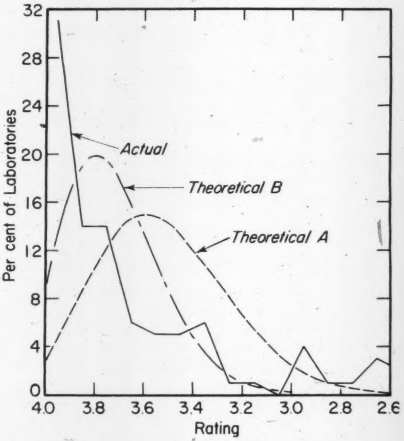


FIG. 9.—Frequency Distribution of Average Laboratory Ratings for All Chemical Tests Reported for Sample No. 6.

The distribution of the ratings for all 10 physical and 10 chemical tests made on sample No. 6, together with two theoretical curves, are presented in Figs. 8 and 9 respectively. These were typical of the distribution obtained on all 12 samples for each test method.

Theoretical curve *A* was based on averages of ten scores where individual scores were assigned as shown in Fig. 7. The standard deviations used are those shown in Table IV which were computed by using all results except obvious blunders. One possible interpretation of the disagreement between curve *A* and the observed frequency distribution of the laboratory scores is in the assumption that all but a small number of laboratories are in better agreement than is reflected in these standard deviations and that the latter are inflated through the inclusion of the excessively discrepant laboratories. This would imply, for example, that some results which fell in this range and were awarded a score of 4 should have had a lower score. Consequently, the proportion of high scores actually awarded was greater than predicted by curve *A*. Curve *B* was derived by recomputing the probabilities for each score, making the arbitrary assumption that the actual standard deviation was 80 per cent of that shown in Table IV. The elimination of the results from about six laboratories with lowest ratings would bring this reduction in the standard deviation. The theoretical distribution of the average scores was calculated and adjusted to 94 laboratories instead of 100, as used for curve *A*. Curve *B* is a better approximation to the observed distribution of average scores and, therefore, tends to support the smaller standard deviations as better estimates of the actual performance of over 90 per cent of the laboratories.

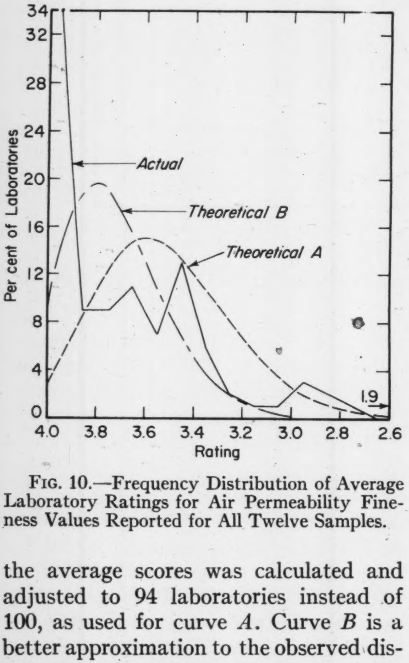


FIG. 10.—Frequency Distribution of Average Laboratory Ratings for Air Permeability Fineness Values Reported for All Twelve Samples.

In drawing the above conclusions from the frequency distribution of the scores, it should be borne in mind that both theoretical curves are based on the assumption that the scores obtained by any given laboratory for different tests or different samples are statistically independent. An examination of the individual scores reveals, however, that such scores tend to be correlated in a number of cases over the entire sampled population of laboratories. This would indicate that a laboratory tends to maintain its position (as indicated by the scoring system) for different samples on the same test and even, in some cases, for different tests. The disagreement between the theoretical curve *A* and the observed frequency distribution of scores must then be ascribed, in part at least, to those correlations, and this possibility is supported by the still unsatisfactory agreement of the observed distribution of scores with curve *B*.

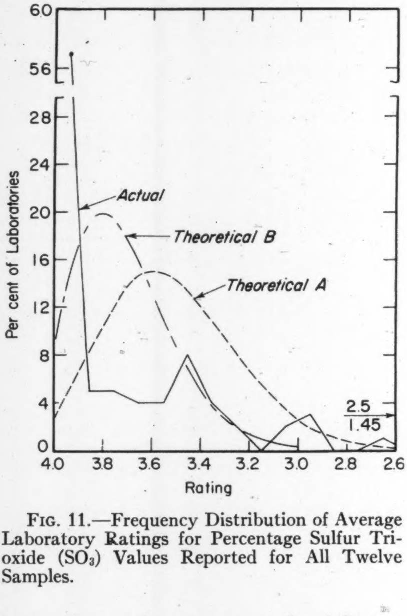


FIG. 11.—Frequency Distribution of Average Laboratory Ratings for Percentage Sulfur Trioxide (SO_3) Values Reported for All Twelve Samples.

But whether the distortion of the frequency distribution of the scores is ascribed primarily to the inflation of the standard deviations through inclusion of a few discrepant laboratories or to the correlation between scores obtained by the same laboratory, in either case the data show the presence of recognizable differences in the performance of the laboratories.

TABLE VII.—FREQUENCY DISTRIBUTIONS OF AVERAGE RATINGS OF ALL LABORATORIES REPORTING PHYSICAL TEST RESULTS.

Tests	a	b	c	d	e	f	g	h	i	j	Total	Average, per cent
Ratings												
4.00 to 3.91	18	17	20	35	27	33	17	21	30	34	252	24.53
3.90 to 3.81	11	17	16	9	9	10	17	12	15	9	125	12.17
3.80 to 3.71	20	6	15	9	9	15	8	13	12	9	116	11.29
3.70 to 3.61	10	9	7	12	12	7	13	9	13	11	103	10.02
3.60 to 3.51	7	10	10	7	7	9	9	5	2	7	73	7.10
3.50 to 3.41	16	10	4	11	13	3	12	14	8	13	104	10.12
3.40 to 3.31	7	5	5	5	7	6	4	7	4	6	56	5.45
3.30 to 3.21	5	12	3	4	3	3	4	8	5	2	49	4.77
3.20 to 3.11	2	6	8	4	3	5	3	4	2	1	38	3.70
3.10 to 3.01	1	2	4	2	2	2	4	1	1	1	20	1.94
3.0 to 2.91	1	1	2	2	3	1	7	2	2	3	24	2.33
2.90 to 2.81	2	1	...	...	2	2	2	2	1	2	14	1.36
2.80 to 2.71	...	2	1	1	...	2	...	1	2	1	10	0.97
2.70 to 2.61	...	...	...	...	2	2	1	...	1	...	6	0.58
2.60 to 2.51	...	...	1	...	1	...	...	1	1	...	4	0.38
2.50 to 2.41	1	1	1	...	...	1	...	3	...	...	7	0.69
2.40 to 2.31	...	1	2	...	1	...	2	...	1	...	7	0.68
2.30 to 2.21	...	...	1	...	...	...	...	...	...	...	1	0.097
2.20 to 2.11	...	1	...	...	...	...	...	...	2	...	3	0.294
2.10 to 2.01	...	1	...	...	...	...	...	...	...	...	1	0.097
2.00 to 1.91	...	...	1	...	...	1	...	...	...	...	2	0.194
1.90 to 1.81	...	...	1	...	...	...	...	...	...	1	2	0.194
1.80 to 1.71	...	...	...	...	...	...	...	...	...	...	...	...
1.70 to 1.61	...	1	...	...	...	...	...	...	...	...	1	0.097
1.60 to 1.51	...	...	...	...	...	1	...	...	...	...	1	0.097
1.50 to 1.41	1	...	...	...	1	...	...	...	...	...	2	0.194
1.40 to 1.31	...	...	...	1	...	...	...	...	...	...	1	0.097
1.30 to 1.21	...	...	1	...	...	...	...	...	...	...	1	0.097
1.20 to 1.11	...	...	...	...	1	...	...	...	...	...	1	0.097
1.10 to 1.01	...	...	...	1	...	...	...	...	...	...	1	0.097
1.00 to 0.91	1	...	...	...	...	...	...	...	...	...	1	0.097
Total	103	103	103	103	103	103	103	103	103	100	1027	99.82

[a] = Normal consistency test.
[b] = Initial setting, Gillmore test.
[c] = Final setting, Gillmore test.
[d] = Autoclave expansion test.
[e] = Air entrainment test.
[f] = Water for air entrainment test.
[g] = Compressive strength, 3 days, test.
[h] = Compressive strength, 7 days, test.
[i] = Water for compressive strength test.
[j] = Air permeability fineness test.

In Figs. 10 and 11 are presented the frequency distributions of the laboratory ratings for one physical test for all 12 samples and for one chemical test for all 12 samples, together with the two theoretical frequency distribution curves. The patterns indicated in both cases are similar to those indicated in Figs. 8 and 9.

These two curves also are typical of the 10 chemical tests and the 10 physical tests performed on each of the 12 samples.

The frequency distributions of the ratings of the laboratories for each of the 10 physical tests for the 12 samples are presented in Table VII, and, for the 10 chemical tests, in Table VIII. The majority of the laboratories had satisfactory ratings, although many had low or very low ratings. As pointed out by Youden,[3] the low ratings of a few laboratories significantly affected the over-all ratings of all laboratories.

Further studies of the frequency distribution of scores are illustrated in Fig. 12. In this figure the frequency distribution of the laboratory ratings for all physical and chemical tests for all samples are presented with the two theoretical curves. It may be noted that more laboratories had ratings of 4.0 to 3.9 for both chemical and physical tests than would be predicted from the theoretical curves *A* or *B*. The number of laboratories with ratings of 3.8 to 3.5 was less and the number of laboratories with very low ratings was more than would be predicted from the theoretical curves. Figures 8 to 11

TABLE VIII.—FREQUENCY DISTRIBUTIONS OF AVERAGE RATINGS OF ALL LABORATORIES REPORTING CHEMICAL TEST RESULTS.

Tests	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Loss on Ignition	Residue	Na_2O	K_2O	Total	Average, per cent
Ratings												
4.00 to 3.91	43	37	61	45	41	57	48	43	27	25	427	45.32
3.90 to 3.81	4	15	14	16	8	5	7	15	5	10	99	10.50
3.80 to 3.71	9	9	...	6	8	5	4	5	7	5	56	5.94
3.70 to 3.61	9	3	5	5	5	4	10	6	6	1	58	6.15
3.60 to 3.51	5	7	5	4	5	4	5	6	6	3	50	5.30
3.50 to 3.41	10	7	4	7	7	8	7	5	3	7	65	6.90
3.40 to 3.31	4	4	2	5	2	4	2	3	2	4	34	3.60
3.30 to 3.21	1	3	...	1	6	2	1	4	...	3	17	1.80
3.20 to 3.11	1	5	3	3	2	...	3	...	3	1	21	2.22
3.10 to 3.01	1	2	...	...	3	2	...	2	1	2	13	1.38
3.00 to 2.91	6	2	1	1	4	3	4	3	2	2	28	2.97
2.90 to 2.81	1	...	1	1	2	...	1	1	...	1	8	0.84
2.80 to 2.71	...	...	1	...	1	...	1	1	1	1	6	0.63
2.70 to 2.61	1	...	...	1	...	1	...	1	...	...	4	0.42
2.60 to 2.51	...	...	...	...	1	...	1	1	...	...	3	0.31
2.50 to 2.41	1	...	1	2	1	3	3	3	2	...	16	1.69
2.40 to 2.31	...	...	...	...	1	...	1	...	1	...	3	0.31
2.30 to 2.21	1	...	...	...	1	...	...	1	...	...	3	0.31
2.20 to 2.11	...	...	...	...	1	...	...	1	1	...	3	0.31
2.10 to 2.01	...	...	1	...	...	...	...	...	...	1	2	0.21
2.00 to 1.91	1	...	...	1	...	...	1	...	...	1	4	0.42
1.90 to 1.81	1	1	...	...	...	...	...	...	...	...	2	0.21
1.80 to 1.71	...	...	1	...	1	...	...	...	...	...	2	0.21
1.70 to 1.61	1	...	...	...	...	...	1	...	...	...	2	0.21
1.60 to 1.51	...	2	...	...	...	...	1	...	...	...	3	0.31
1.50 to 1.41	1	...	...	...	...	1	...	...	...	...	2	0.21
1.40 to 1.31	...	...	...	1	1	1	...	...	...	...	3	0.31
1.10 to 1.01	...	...	1	...	...	...	...	...	...	...	1	0.10
1.00 to 0.91	...	...	1	...	...	...	...	...	...	...	1	0.10
0.90 to 0.81	...	...	2	...	...	...	...	...	...	...	2	0.21
0.80 to 0.71	...	...	...	...	1	...	...	...	...	...	1	0.10
0.70 to 0.61	...	...	...	1	...	...	...	...	...	...	1	0.10
0.60 to 0.51	...	...	...	...	...	...	...	...	...	...	...	...
0.50 to 0.41	...	...	...	1	...	...	...	...	...	...	1	0.10
0.40 to 0.31	...	...	...	...	...	...	1	...	...	...	1	0.10
Total	101	101	101	101	101	101	101	101	67	67	942	99.80

similarly indicate departures from the theoretical curves.

These tables and curves show that an improvement in the ratings of a relatively small proportion of the laboratories would result in a considerable improvement in the standard deviations of all tests.

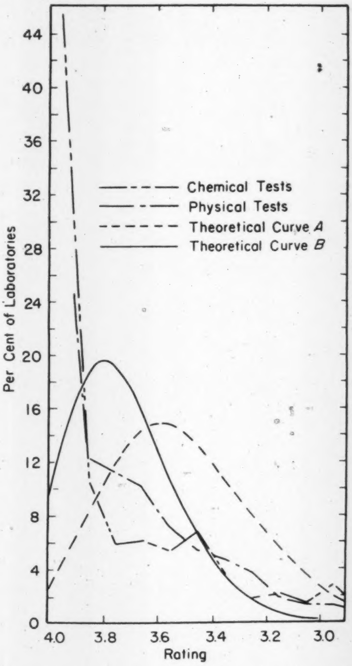


FIG. 12.—Frequency Distribution of Average Laboratory Ratings for All Physical Tests and All Chemical Tests Reported for All Twelve Samples.

On the basis of the data obtained in this program, it might be suggested that any laboratory that consistently has a rating of less than 3.5 by this rating system might well look into the possible causes for disagreement with the majority of laboratories.

As indicated earlier, all data were included in the tabulations and calculations except the obvious errors. Evidence has been presented that some of the data included were subject to extreme systematic errors and should have been excluded. It therefore appeared desirable to use another method for rating the laboratories.

Six large cement manufacturing companies were asked to advise NBS as to the tolerance limits used by them for each of the various physical and chemical tests in their interlaboratory test programs. These six tolerances, averaged for each of the tests, were used to re-evaluate the performance of all the participating laboratories. The tolerances were applied to the averages for the values reported by all the laboratories for each of the tests.

Table IX lists for each of the physical and chemical tests of the 12 samples, the average standard deviations, the averages of the tolerances suggested by the 6 laboratories, the percentage of laboratories passing these tolerances, and also the values for $a/2$ (see Table V). It may be noted that the values for the average standard deviations developed in this program and the average tolerances of the 6 cement laboratories were very similar in the case of both the physical and chemical tests. The percentage of laboratories passing the tolerances was generally higher in chemical than in physical tests.

The values for a presented in Table V represented the spread of 68 per cent of the results closest to the 45-deg line. It has previously been shown that these values are a measure of the random errors of the test methods. With normal distribution, the results of this same number of laboratories, 68 per cent, should be within plus or minus one standard deviation of the mean, and half of this value, or $a/2$, should correspond to the standard deviation of the test method with the effect of laboratory bias eliminated. Values of $a/2$ presented in Table IX for the various tests may be compared with the average standard deviation values obtained in this test program for the physical and chemical tests of the various cements and to the average tolerances of the 6 laboratories. It may be noted that the values for the average standard deviations for the different tests were, in most instances, 2 to 3 times the corresponding values for $a/2$, the standard deviation values attributable to the precision of the test methods.

TABLE IX.—A COMPARISON OF THE STANDARD DEVIATION VALUES ATTRIBUTABLE TO THE TEST METHODS, $a/2$,[a] THE STANDARD DEVIATION VALUES FOR THE 12 SAMPLES, AND THE TOLERANCE VALUES PERMITTED BY 6 LABORATORIES WHO REGULARLY CONDUCT INTERLABORATORY TESTS.

	$\frac{a}{2}$	Average Standard Deviation, 12 Samples	Permitted Tolerance of 6 Laboratories, average	Per Cent Laboratories Passing Tolerance of 6 Laboratories
PHYSICAL TESTS				
Initial setting, hr	0.36	0.52	±0.50	71
Final setting, hr	0.40	0.86	±0.66	62
Expansion, per cent	0.012	0.03	±0.06	95
Air entrainment, per cent	0.65	1.40	±1.25	71
Compressive strength, 3 days, psi	128	218	±10 %	76
Compressive strength, 7 days, psi	157	285	±10 %	83
Fineness, Blaine, sq cm per g	81	146	±100	71
CHEMICAL TESTS				
SiO_2, per cent	0.085	0.24	±0.21	76
Al_2O_3, per cent	0.130	0.36	±0.22	69
Fe_2O_3, per cent	0.035	0.14	±0.12	89
CaO, per cent	0.125	0.47	±0.27	70
MgO, per cent	0.070	0.20	±0.21	83
SO_3, per cent	0.035	0.11	±0.11	87
Ignition loss, per cent	0.090	0.16	±0.19	88
Insoluble residue, per cent	0.030	0.09	±0.11	91
Na_2O, per cent	0.010	0.04	±0.04	85
K_2O, per cent	0.025	0.05	±0.04	79

[a] See Table V.

ADDITIONAL TESTS

At various times during the program, the laboratories were asked to perform additional tests. Some of these were by ASTM methods, such as the tensile briquette test (C 109 – 58), Vicat time of setting (C 191 – 58), Wagner turbidimeter for fineness (C 115 – 58), heat of hydration (C 186 – 58), and false setting (C 359 – 56 T).[4] The sulfate resistance test used was one that was under study and was not an ASTM test method. The standard deviations and coefficients of variation for these tests are of about the same order of magnitude as those obtained with the other tests performed on all 12 samples (see Table IV).

Several other variations in procedure were introduced into the test program in order to determine the effect of certain variables.

With two separate pairs of samples, namely, Nos. 3 and 4 and Nos. 12 and 11, half the laboratories performed their chemical tests on cement samples that had been more thoroughly blended and then packaged in sealed vials, while the other half of the laboratories performed their chemical tests on portions of cement taken from the 12-lb samples used for all other tests.

---

[4] These methods of test appear in the 1958 Book of ASTM Standards, Part 4.

With one pair of samples, Nos. 7 and 8, half of the laboratories were asked to make strength cubes using regular tap water and to store the cubes in the usual manner until time for breaking, while the other half used distilled water in making the cubes, after which the cubes were stored in a moist plastic bag in the moist cabinet until time for breaking.

Another of the variables in this program was the storage of the samples previous to distribution. It would be desirable for a laboratory, for example, to blend and package the samples during the winter season when the employees are not so busy. Half of lots 3 and 4 were therefore stored in sealed metal drums and half in the shipping packages on a platform skid, in a store room. These lots were shipped out 10 months later as samples Nos. 12 and 11 respectively.

TABLE X.—FREQUENCY DISTRIBUTION OF COMPOUND COMPOSITION VALUES COMPUTED FROM CHEMICAL ANALYSIS VALUES REPORTED FOR SAMPLE NO. 9.

C_3A, per cent	Number of Laboratories	C_3S, per cent	Number of Laboratories	C_2S, per cent	Number of Laboratories	C_4AF, per cent	Number of Laboratories
1.1 to 2.0........	3	33.1 to 34.0	1	16.1 to 17.0	1	12.1 to 13.0	1
2.1 to 3.0........	1	34.1 to 35.0	0	...	0	13.1 to 14.0	47
3.1 to 4.0........	0	35.1 to 36.0	2	24.1 to 25.0	1	14.1 to 15.0	45
4.1 to 5.0........	0	36.1 to 37.0	1	25.1 to 26.0	1	15.1 to 16.0	1
5.1 to 6.0........	3	37.1 to 38.0	3	26.1 to 27.0	5	16.1 to 17.0	0
6.1 to 7.0........	21	38.1 to 39.0	7	27.1 to 28.0	1	17.1 to 18.0	0
7.1 to 8.0........	48	39.1 to 40.0	11	28.1 to 29.0	6	18.1 to 19.0	0
8.1 to 9.0........	16	40.1 to 41.0	14	29.1 to 30.0	16	19.1 to 20.0	1
9.1 to 10.0......	3	41.1 to 42.0	18	30.1 to 31.0	24		
		42.1 to 43.0	18	31.1 to 32.0	12		
		43.1 to 44.0	5	32.1 to 33.0	14		
		44.1 to 45.0	5	33.1 to 34.0	6		
		45.1 to 46.0	3	34.1 to 35.0	3		
		46.1 to 47.0	3	35.1 to 36.0	3		
		47.1 to 48.0	2	36.1 to 37.0	1		
		...					
		...					
		...					
		...					
		...					
		58.1 to 59.0	1				

The variables of (1) vial *versus* bulk samples, (2) tap *versus* distilled water, and (3) skid *versus* drum storage were examined by alloting 50 of the laboratories to one choice and the other 50 laboratories to the other choice. Consequently, in looking for an effect of any of the three pairs of alternatives listed above, the averages and standard deviations of 50 laboratories using one choice were compared with the averages and standard deviations of the other 50 laboratories using the other choice. These averages, standard deviations, and coefficients of variation are presented in Appendix III. It was concluded, from a study of the data, that in almost all cases, changes in these variables did not produce differences which were statistically significant. Additional data and more precise test procedures may throw further light on the effect of these variables.

PRACTICAL IMPORTANCE OF PRECISE CEMENT TESTING

Precise measurements of the physical and chemical properties of cement are desirable for both manufacturing control and acceptance testing, as well as for research on the effect of these properties of the cement on the properties of concretes made with cements of different composition. For example, the compound composition calculated from the chemical analysis is one of the factors used to distinguish between types I, II, IV, and V cements. As an illustration of the effect of chemical analysis on the calculated compound composition, Table X has been prepared. These compound-composition values were computed from the chemical analyses of sample No. 9 reported by 95 of the laboratories participating in this program. The frequency distributions of the calculated percentages of C_3A, C_3S, C_2S, and C_4AF are presented in Table X. Four of the laboratories would have considered sample No. 9 a type V cement (less than 5.0 per cent C_3A); 19 would have considered it a type I cement (more than 8 per cent C_3A); and the other laboratories would have considered it a type II cement. In view of the fact that present cement specifications permit up to 3.0 per cent SO_3 when the C_3A is greater than 8.0 per cent and only 2.5 per cent SO_3 when the C_3A is 8.0 per cent or less, 19 of the laboratories would have permitted 3.0 per cent SO_3, whereas the others would have limited the SO_3 to 2.5 per cent. If estimates were made of probable heat of hydration or sulfate resistance from chemical analyses varying as much as those reported in this program, these estimates would not be of very great value.

Similarly, the wide range in results obtained in physical and chemical testing of cement might lead to controversies between the manufacturer and purchaser and might give a false impression of the properties of the cement used in a concrete structure.

Greater precision in test methods and laboratory technique would greatly reduce the margin of safety manufacturers must employ to insure that their product will meet specifications when tested by some other laboratory.

OPERATION OF A CONTINUING STANDARD REFERENCE SAMPLE PROGRAM

When the use of standard cement samples was proposed to ASTM Committee C-1 in 1957, it was pointed out that a continuing Standard Sample program would give the participating laboratories a better means of evaluating their performance and would eventually contribute to the improvement of cement testing.

A number of details were necessary for this preliminary study, which would not be required in a Standard Sample program. From the data reported in this study, the results of certain laboratories were consistently close to the means for all 103 laboratories, and the results of the others were about equally distributed around the means. Ten or 15 laboratories could be selected as referees from among the research, producer, commercial, State and Federal laboratories who have qualified by obtaining acceptable results. These laboratories could be used as a group to develop an average for each test for each sample. Since the chosen laboratories may obtain erratic results once in a while, provisions should be made to exclude values which appear to be out of line.

Cements for the standard samples would be carefully blended, then packaged and checked for uniformity, and mailed to those laboratories desiring to purchase them. Pairs of samples would be made available every two months, as in the program reported here, and on an annual basis.

The selected laboratories previously described would be requested to act as referee laboratories and submit their test results very promptly. The results of these referee laboratories would be averaged, and plots would be prepared using their means as the intersecting lines for scatter diagrams. As the results of those participating were received, they would be plotted on this diagram and, within 30 days or when all results were received, the scatter diagrams would be reproduced and distributed. The laboratories would immediately be able to see how their results compared with the accepted values and with those of all the other laboratories. The comparison would indicate to the participant where remedial action was necessary. A rating system such as described previously could also be adopted. Since the average values would appear on the scatter diagram, tolerances could be set up similar to those shown in Table IX and, with these two known, a laboratory could easily rate itself as was shown in Fig. 7. The rating would be a useful index for each laboratory to assess its conformance with a selected group of laboratories in the testing of cement.

Of course the ability to make these tests correctly is not a guarantee that a laboratory will make all other tests carefully or according to specification, but it certainly is a step in the right direction. If two laboratories were not in agreement in a given case, it would be possible to check their results on previous Standard Reference Samples to see if consistent differences were obtained. Purchasers of testing services may then assure themselves that the laboratories testing cement for them have acceptable ratings.

Laboratories whose results are not in agreement with the accepted values could obtain special attention and assistance from the Cement Reference Laboratory in their regular inspection service.

Scatter diagrams, such as those developed for this program, together with the participation of a large number of laboratories in a program, should offer the various working committees and sponsoring committees of ASTM Committee C-1 information as to whether or not the specification test methods are sufficiently clear and concise that operators can readily follow them.

SUMMARY

As a result of the tests by 103 laboratories on 12 samples of cement, it has been demonstrated that the use of paired samples, with graphic presentation as suggested by Youden, enables an appraisal of systematic laboratory errors and of the precision of the various tests. It was found that the standard deviation for many of the chemical and physical tests for cement was inflated by large differences between laboratories. The average standard deviations for the different tests on the 12 samples were usually about 2 to 3 times the estimated standard deviation attributable to the random errors of measurement associated with test methods.

There was a wide spread in the test results reported for both the physical and chemical tests. When the scores obtained by each laboratory were averaged over several samples or several tests, the number of laboratories obtaining high averages was larger than would be anticipated from a theoretical averaging of independent scores; and the same situation prevailed for the number of laboratories obtaining very low average scores.

The average values for the standard deviation for the different tests of this program were approximately the same as the corresponding average tolerance values of 6 laboratories who regularly conduct interlaboratory tests. The percentage of participants meeting the tolerance requirements of these 6 laboratories ranged from 62 per cent to 95 per cent for the different tests.

Variables introduced into the program, such as (1) use of distilled water *versus* tap water for making cubes, (2) storage of the cement samples for 10 months in shipping bags in a storeroom or in sealed metal drums prior to distribution, and (3) special blending and packaging of chemical samples, did not have a statistically significant effect on average test values or the coefficient of variation of these values. The use of referee chemical methods did not result in better concordance than was obtained with the use of optional chemical methods.

A rating system for individual laboratories was developed which was based on the various test results reported by all the laboratories. Such a rating system, together with the scatter diagrams for each of the tests, enables a laboratory to evaluate its work and take remedial action where necessary.

Because of the apparent need for greater concordance in the testing of cement and because the preliminary reference sample program offered a satisfactory means for evaluating test procedures and laboratories, it has been proposed that a Cement Reference Sample program be established on a continuing basis.

APPENDIX I

LIST OF PARTICIPATING LABORATORIES

Alabama Highway Dept., Montgomery, Ala.
Alpha Portland Cement Co., Martins Creek, Pa., Birmingham, Ala.
Arizona Highway Dept., Phoenix, Ariz.
Ash Grove Lime & Portland Cement Co., Chanute, Kans.
Browser-Morner Testing Labs., Dayton, Ohio.
Calaveras Cement Co., San Andreas, Calif.
California Highway Dept., Sacramento, Calif.
California Portland Cement Co., Colton, Calif.
Colorado Highway Department, Denver, Colo.
Consolidated Cement Co., Fredonia, Kans.
E. L. Conwell & Co., Philadelphia, Pa.
Coplay Cement Mfg. Co., Coplay, Pa.
Corps of Engineers, Dallas, Tex., Jackson, Miss., Marietta, Ga.
Dewey Portland Cement Co., Dewey, Okla., Davenport, Iowa.
Dragon Cement Co., Northampton, Pa.
Diamond Portland Cement Co., Middle Branch, Ohio.
Froehling & Robertson, Inc., Richmond, Va.
General Portland Cement Co., Houston, Tex., Tampa, Fla.
Georgia Highway Dept., Atlanta, Ga.
Giant Portland Cement Co., Egypt, Pa.
Glens Falls Portland Cement Co., Glens Falls, N. Y.
Haller Testing Laboratories, Inc., Plainfield, N. J.
Halliburton Portland Cement Co., Corpus Christi, Tex.
Hercules Cement Corp., Nazareth, Pa.
Huron Portland Cement Co., Alpena, Mich.
Ideal Cement Co., Fort Collins, Colo., Baton Rouge, La.
Illinois Highway Dept., Springfield, Ill.
Indiana Highway Dept., Indianapolis, Ind.
Iowa Highway Dept., Ames, Iowa.
Kansas Highway Dept., Manhattan, Kans.
Keystone Portland Cement Co., Bath, Pa.
Kosmos Portland Cement Co., Kosmosdale, Ky.
Lehigh Portland Cement Co., Allentown, Pa., Birmingham, Ala.
Lone Star Cement Co., Hudson, N. Y., Dallas, Tex.
Louisiana Highway Dept., Baton Rouge, La.
Louisville Cement Co., Speed, Ind.
Maine Highway Dept., Orono, Me.
Marquette Cement Co., Chicago, Ill., Brandon, Miss.

Maryland State Roads Commission, Baltimore, Md.
Medusa Portland Cement Co., Wampum, Pa.
Manitowoc Portland Cement Co., Manitowoc, Wis.
Michigan State Highway Dept., Lansing, Mich.
Minnesota Highway Dept., St. Paul, Minn.
Missouri Highway Commission, Jefferson City, Mo.
Missouri Portland Cement Co., St. Louis, Mo., Independence, Mo.
The Monarch Cement Co., Humboldt, Kans.
Monolith Portland Cement Co., Monolith, Calif.
National Bureau of Standards, Allentown, Pa., Denver, Colo., San Francisco, Calif., Seattle, Wash., Washington, D. C.
National Cement Co., Ragland, Ala.
National Portland Cement Co., Bethlehem, Pa.
U. S. Naval Civil Engineering Laboratory, Port Hueneme, Calif.
Nazareth Portland Cement Co., Nazareth, Pa.
Nebraska Highway Dept., Lincoln, Nebr.
New Jersey Highway Dept., Trenton, N. J.
North Carolina Highway Dept., Raleigh, N. C.
The H. C. Nutting Co., Cincinnati, Ohio.
Ohio Highway Dept., Columbus, Ohio.
Olympia Portland Cement Co., Ltd., Bellingham, Wash.
Oregon Highway Dept., Salem, Ore.
Pacific Cement & Aggregates, Inc., Davenport, Calif.
Peerless Cement Co., Detroit, Mich., Port Huron, Mich.
Penn-Dixie Cement Corp., Nazareth, Pa., West Winfield, Pa.
Pennsylvania Highway Dept., Harrisburg, Pa.
Permanente Cement Co., Permanente, Calif., Lucerne Valley, Calif.
Pittsburgh Coke & Chemical Co., Neville Island, Pa.
Pittsburgh Testing Laboratory, Pittsburgh, Pa.
Portland Cement Assn. Chicago, Ill.
Bureau of Public Roads, Washington, D. C.
Raymond G. Osborne Laboratories, Inc., Los Angeles, Calif.
U. S. Bureau of Reclamation, Denver, Colo.
Standard Lime & Cement Co., Martinsburg, W. Va.
Shilstone Testing Laboratory, New Orleans, La.
Southern Cement Co., Birmingham, Ala.
Southwestern Portland Cement Co., Fairborn, Ohio, Victorville, Calif., El Paso, Tex.
Texas Highway Dept., Austin, Tex.
The Thompson & Lichtner Co., Inc., Brookline, Mass.
Toledo Testing Laboratory, Toledo, Ohio.
Twin City Testing & Engineering Lab., St. Paul, Minn.
Universal Atlas Cement Co., Gary, Ind., Northampton, Pa.
Volunteer Portland Cement Co., Knoxville, Tenn.
The Whitehall Cement Mfg. Co., Cementon, Pa.
West Virginia Highway Dept., Morganstown, W. Va.
Wisconsin Highway Dept., Madison, Wis.

APPENDIX II

AVERAGES OF RESULTS REPORTED BY ALL LABORATORIES

Cement Samples	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10	No. 11	No. 12
Water consistency, per cent	24.6	25.5	27.0	25.9	24.9	27.1	28.7	24.5	24.4	25.6	26.0	25.9
Initial setting, Gillmore, hr	3.19	3.45	2.94	3.26	...	...	2.85	3.04	3.66	4.76	3.23	2.92
Final setting, Gillmore, hr	5.43	5.48	4.97	5.31	...	...	4.81	5.10	5.84	7.49	5.37	4.98
Expansion, per cent	0.14	0.13	0.06	0.08	0.03	0.04	0.02	0.18	0.03	0.03	0.09	0.06
Air entrainment, per cent	7.2	8.5	8.1	16.3	9.8	11.8	7.0	19.3	11.1	8.8	16.7	9.5
Water-air entrainment, per cent	70.3	69.5	70.0	60.5	68.5	67.0	70.4	57.5	67.1	67.8	58.8	68.5
Compressive strength, 3 days, psi	2327	2848	2764	2207	1889	2208	2675	1801	1868	2285	2292	2771
Compressive strength, 7 days, psi	3759	3675	4245	3617	2674	3645	4794	2481	2780	3389	3734	4302
Water for compressive strength, per cent	49.1	47.9	48.7	47.4	47.4	48.9	48.9	45.8	46.9	47.3	46.6	48.0
Fineness, Blaine, sq cm per g	3283	3442	3820	3107	3573	4564	4588	3147	3410	2949	3139	3875
SiO_2, per cent	21.08	21.73	21.74	21.70	21.24	23.22	20.76	22.25	21.65	23.48	21.65	21.68
Al_2O_3, per cent	6.83	4.95	5.87	5.88	5.47	8.13	5.60	5.12	5.76	3.67	5.90	5.90
Fe_2O_3, per cent	2.48	2.88	2.82	2.73	4.76	2.58	2.50	2.36	4.63	4.44	2.69	2.75
CaO, per cent	63.12	61.90	63.97	65.02	61.98	55.35	64.24	62.44	63.00	64.39	64.96	63.93
MgO, per cent	2.93	3.81	1.83	1.36	2.42	3.89	1.97	3.59	1.15	0.76	1.35	1.80
SO_3, per cent	1.80	2.79	1.82	1.78	1.92	2.43	2.84	2.18	1.87	1.80	1.78	1.82
Ignition loss, per cent	0.75	0.79	1.18	0.79	1.38	2.63	1.26	0.86	1.05	0.76	0.82	1.25
Residue, per cent	0.16	0.25	0.15	0.18	0.18	0.53	0.21	0.17	0.14	0.14	0.13	0.13
Na_2O, per cent	0.36	0.30	0.19	0.14	0.24	0.10	0.15	0.30	0.15	0.50	0.17	0.21
K_2O, per cent	0.70	1.00	0.74	0.75	0.59	0.25	0.76	1.08	0.82	0.21	0.77	0.76
Mn_2O_3, per cent	...	...	...	...	0.09	0.64	...	...	...	...	...	...
Sulfide sulfur, per cent	...	...	...	...	0.01	0.59	...	...	...	...	...	...
False setting, initial, mm	...	...	...	...	...	...	...	...	...	...	31	33
False setting, final, mm	...	...	...	...	...	...	...	...	...	...	20	19
Tensile strength, 3 days, psi	...	...	352	317	...	...	...	...	...	...	...	...
Tensile strength, 7 days, psi	...	...	424	395	...	...	...	...	...	...	...	...
Water for tensile strength, per cent	...	...	10.8	10.8	...	...	...	...	...	...	...	...
Vicat setting, hr	...	...	...	...	2.66	2.97	...	...	...	...	...	...
Fineness, Wagner, sq cm per g	...	...	...	...	...	...	2547	1739	...	...	...	...
Retained on No. 325 sieve, g	...	...	...	...	...	...	0.01	0.10	...	...	...	...
Sulfate resistance expansion, 14 days, per cent	...	...	...	...	...	...	...	...	0.005	0.002	...	...
Sulfate resistance expansion, 28 days, per cent	...	...	...	...	...	...	...	...	0.018	0.009	...	...
Heat of hydration, 7 days, cal per g	...	...	...	...	...	...	...	...	69	64	...	...
Heat of hydration, 28 days, cal per g	...	...	...	...	...	...	...	...	82	77	...	...

APPENDIX III

AVERAGE RESULTS, WITH COMPUTED STANDARD DEVIATIONS AND COEFFICIENTS OF VARIATION FOR CHEMICAL ANALYSIS OF SAMPLES NO. 3 AND NO. 4.[a]

	Sample No. 3						Sample No. 4					
	Vial			Bulk			Vial			Bulk		
	Average, per cent	Standard Deviation	Coefficient of Variation, per cent	Average, per cent	Standard Deviation	Coefficient of Variation, per cent	Average, per cent	Standard Deviation	Coefficient of Variation, per cent	Average, per cent	Standard Deviation	Coefficient of Variation, per cent
Silicon dioxide (SiO_2)	21.73	0.183	0.84	21.75	0.182	0.84	21.71	0.215	0.99	21.69	0.174	0.80
Aluminum oxide (Al_2O_3)	5.88	0.241	4.10	5.86	0.315	5.38	5.88	0.165	2.81	5.90	0.310	5.25
Ferric oxide (Fe_2O_3)	2.84	0.266	9.37	2.80	0.171	6.11	2.74	0.257	9.38	2.73	0.138	5.05
Calcium oxide (CaO)	63.91	0.447	0.70	64.03	0.280	0.44	65.02	0.348	0.54	64.98	0.353	0.54
Magnesium oxide (MgO)	1.84	0.138	7.50	1.81	0.156	8.62	1.36	0.162	11.91	1.34	0.106	7.91
Sulfur trioxide (SO_3)	1.82	0.071	3.90	1.81	0.104	5.75	1.76	0.093	5.28	1.79	0.083	4.64
Loss on ignition	1.16	0.133	11.47	1.20	0.141	11.75	0.80	0.163	20.38	0.80	0.164	20.50
Insoluble residue	0.13	0.049	37.69	0.17	0.110	64.70	0.18	0.165	91.67	0.14	0.108	77.14
Sodium oxide (Na_2O)	0.18	0.024	13.33	0.19	0.031	16.32	0.14	0.032	22.86	0.15	0.022	14.67
Potassium oxide (K_2O)	0.75	0.038	5.28	0.73	0.053	7.26	0.74	0.045	6.08	0.76	0.023	3.03

[a] Half of the laboratories made tests on specially blended samples of cement packaged in sealed vials, whereas the other laboratories used portions of cement from the bulk samples for chemical analysis.

AVERAGE RESULTS, WITH COMPUTED STANDARD DEVIATIONS AND COEFFICIENTS OF VARIATION FOR COMPRESSIVE STRENGTH TESTS OF SAMPLES NO. 7 AND NO. 8.[a]

	Sample No. 7						Sample No. 8					
	Distilled			Tap			Distilled			Tap		
	Average, psi	Standard Deviation	Coefficient of Variation, per cent	Average, psi	Standard Deviation	Coefficient of Variation, per cent	Average, psi	Standard Deviation,	Coefficient of Variation, per cent	Average, psi	Standard Deviation	Coefficient of Variation, per cent
1 day	2644	190	7.2	2708	204	7.5	...	...	...	...	...	...
3 days	4796	262	5.5	4777	259	5.4	1799	139	7.7	1788	139	7.8
7 days	...	...	...	...	...	...	2442	161	6.6	2429	185	7.6

[a] Half the laboratories made the test by regular ASTM procedure, while the other laboratories used distilled water for making cubes, after which the cubes were stored in a moist plastic bag in the moist cabinet until time for breaking.

[Continued on page 1154.]

AVERAGE RESULTS, WITH COMPUTED STANDARD DEVIATIONS AND COEFFICIENTS OF VARIATION FOR ALL THE PHYSICAL AND CHEMICAL TESTS ON SAMPLES NO. 11 AND NO. 12.[a]

	Sample No. 11						Sample No. 12					
	Skid			Drum			Skid			Drum		
	Average	Standard Deviation	Coefficient of variation	Average	Standard Deviation	Coefficient of Variation	Average	Standard Deviation	Coefficient of Variation	Average	Standard Deviation	Coefficient of Variation
Water consistency, per cent	25.4	0.504	1.99	25.8	0.543	2.10	25.6	1.006	3.93	26.0	0.550	2.12
Initial setting, Gillmore, hr	3.21	0.251	7.82	3.24	0.395	12.19	2.91	0.418	14.35	2.93	0.485	16.55
Final setting, Gillmore, hr	5.47	0.716	13.09	5.27	0.654	12.41	4.88	0.584	11.98	5.07	0.681	13.43
Expansion, per cent	0.071	0.018	24.7	0.076	0.019	25.0	0.051	0.013	24.6	0.059	0.018	35.3
Air entrainment, per cent	16.6	1.21	7.26	16.8	1.80	10.72	9.46	1.17	12.33	9.59	1.37	14.31
Water-air entrainment, per cent	58.3	2.39	4.11	59.3	3.13	5.27	68.6	2.11	3.07	68.4	2.08	3.04
Compressive strength 3 days, psi	2346	184	7.84	2229	222	9.96	2788	292	10.47	2767	207	7.48
Compressive strength 7 days, psi	3787	321	8.48	3674	251	6.83	4352	292	6.71	4256	340	7.99
Water for compressive strength, per cent	46.2	0.993	2.15	46.9	1.12	2.40	47.9	1.27	2.64	48.0	0.912	1.90
Fineness, Blaine, sq cm per g	3153	66.0	2.09	3125	180	5.76	3892	121	3.11	3859	94	2.44
False setting, initial mm	34.15	3.21	9.40	27.71	4.81	17.36	34.09	5.87	17.22	33.60	5.56	16.55
False setting final mm	22.21	4.93	22.20	17.65	4.97	28.16	12.88	5.84	45.34	25.28	6.42	25.40
SiO_2, per cent	21.66	0.305	1.41	21.64	0.378	1.75	21.65	0.310	1.43	21.70	0.176	0.81
Al_2O_3, per cent	5.88	0.18	3.06	5.92	0.251	4.24	5.91	0.228	3.86	5.89	0.248	4.21
Fe_2O_3, per cent	2.71	0.071	2.60	2.68	0.119	4.44	2.74	0.114	4.16	2.76	0.063	2.28
CaO, per cent	64.95	0.419	0.65	65.00	0.495	0.76	63.96	0.367	0.57	63.91	0.460	0.72
MgO, per cent	1.34	0.127	9.48	1.39	0.220	15.83	1.79	0.231	12.91	1.82	0.185	10.17
SO_3, per cent	1.78	0.059	3.33	1.78	0.059	3.33	1.83	0.833	4.55	1.81	0.081	4.47
Ignition loss, per cent	0.86	0.104	12.09	0.77	0.097	12.58	1.28	0.140	10.94	1.23	0.129	10.49
Residue, per cent	0.13	0.060	45.76	0.13	0.063	48.54	0.13	0.056	43.00	0.13	0.472	36.31
Na_2O, per cent	0.17	0.020	11.88	0.17	0.042	24.53	0.21	0.043	20.67	0.21	0.022	10.52
K_2O, per cent	0.77	0.031	3.99	0.76	0.056	7.33	0.76	0.050	6.61	0.76	0.037	4.80

---

[a] Half of each of these samples packaged for shipment were stored 10 months in sealed metal drums and the other half were stored 10 months on a platform skid in a storeroom.

FUNDAMENTAL CHARACTERISTICS OF PULVERIZED COAL FLY ASHES*

By L. John Minnick[1]

Synopsis

Data are presented relating to the basic properties and characteristics of fly ash with emphasis being placed on information unavailable in previous publications. The heterogeneous property of fly ash is considered and a critical analysis is made of the component parts of the material including a study of structure and mineral placement. The investigation utilizes chemical and physical tests based on X-ray diffraction and fluorescent spectrographic analyses, microscopic examination, and a few DTA analyses. Pozzolan reactives are considered using several curing conditions in mixtures made with high calcium and dolomitic lime. The study is coordinated with investigations being carried out in ASTM committees and also includes some information obtained from an extensive questionnaire that was sent to the fly ash producers regarding types of coal, methods of burning, and methods of collection.

Almost from the time that fly ash was first produced from the combustion of pulverized coal, a number of investigations were undertaken to find commercial uses for this material. These efforts have resulted in a variety of applications based upon its engineering properties. Fly ash is being used in substantial quantities in concrete, structural building units, plasters, mortars, asphaltic and plastic compositions, ceramic products, and as an integral part of road bases. Recently fly ash has been separated into several products, namely, a siliceous magnetite or "iron fraction" and a quality controlled or beneficiated pozzolan. Some progress has also been made in the conversion of fly ash into light weight aggregate.

Many articles have been published on the utilization of fly ash which describe a variety of compositions, mixtures, and end products (**1**).[2] Some of these studies are concerned with chemical and physical properties of fly ashes—a few consider basic reaction with alkali and lime (**2**). The U. S. Bureau of Mines has recently made a comprehensive summary of over 300 samples of ash which includes chemical analysis of fly ashes and pertinent information on the coal from which the ash is produced (**3**). Much of the published literature makes comparisons between different samples or sources of fly ash and in general does not study the effect of altering the ingredients within a given fly ash. It is well known that considerable variation exists between sam-

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26 1959.

[1] Vice-president in Charge of Research, G. & W. H. Corson, Inc., Plymouth Meeting, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—DESCRIPTION OF COAL USED TO PRODUCE FLY ASH.

Fly Ash Number	Designation Series	Location of Mine	Type of Mine	Ash, per cent	Fixed Carbon, per cent	Volatile, per cent	Thermal Conductivity Units	Grindability, per cent	Coal Fineness When Burned, Passing No. 200 Sieve, per cent	Type of Firing	Fly Ash Collecting System
	LF	Low (0 to 6 Per Cent) Ignition—More Than 88 Per Cent Passing No. 325 Sieve									
A........		West Ky.	Deep and strip	14	42.87	33.13	11 300	47	70	Horizontal intervane	Cottrell precipitation
S-1.......		West Ky.		14	42.87	33.13	11 300	47	70	Horizontal intervane	Cottrell precipitation
X-16.....		Fulton Co.	Strip	9.25	...	...	10 500	...	...	Pulverized fuel	Electrostatic precipitation
		Central Co.	Shaft	10.40	43	32	10 428	...	75	Vertical downward	
S-2.......		Central Co.	Shaft	9.25 10.40	... 43	... 32	10 500 10 428	... ...	... 75	Vertical downward	Precipitation
S-3.......		Central Pa., bituminous	Deep and strip	7.9	51.8	32.8	12 940	...	75	Multitube burner	Cyclone
S-4.......		Pittsburgh Seam	Deep mine	8.4	55.4	36.2	13 838	62.2 to 74.7	82	Horizontal burner	Electrostatic precipitation
S-5.......		Muskingum, Ohio	Surface	15.16	39.78	36.73	10 794	56.7	75 to 90	Vertical fixed by force	Mechanical aerotic tubular dust collector
S-6.......		Ohio, bituminous	Strip	3.2	11.0	...	...	...	...	Pulverized fuel	HydroVac with aerotic
S-7.......		W. Va.	Strip	12.2	58.3	22.5	11 700	...	85 to 90	Flare	Cottrell
S-8.......		Awkright	Strip	13.2	50.8	31.0	12 050	62	75	...	Electrostatic precipitation
S-9.......		Randolph and St. Clair	...	9.0	...	...	11 300	...	75	Pulverized fuel horizontal	Electrostatic precipitation
		Ind.	...	7.66	...	...	11 800	...	75	...	Electrostatic precipitation
		West Ky.	...	9.43	...	...	11 806	...	75	...	Electrostatic precipitation
S-10......		85 per cent Freeport 15 per cent Bakerstown	Deep strip	20.0	47.9	24.2	10 722	55 to 95	80	...	...
S-11......		West Pa. Fairmount	Strip	11.0	47.0	34.0	12 300	60	80	Multiple inner tube burner	Vacuum system
S-17......		Western Pa.	Deep and strip	15.0	50 to 52	33 to 35	12 400	...	75	Pressured direct fired	Mechanical and electrostatic precipitation
		Western Pa.	Strip	19.0	47 to 50	31 to 34	11 750	...	75	Exhauster direct fired	Electrostatic precipitation

	LC	LOW (0 TO 6 PER CENT) IGNITION—LESS THAN 88 PER CENT PASSING NO. 325 SIEVE								
B........		East Tenn. Kentucky	Strip	10.9	52.9	34.5	12 423	52 to 56	65 to 70	Tangential	Mechanical
C........		...	Deep and strip	...	...	...	...	...	75	Vertical (roof down) pulverized coal	Electrostatic precipitation
D........		Pittsburg W. Va.	Seam	9.0	51.0	40.0	13 800	...	70	...	...
F........		W. Pa. and W. Va.	Deep mine	7.50	61.0	28.0	13 500	75	20	Multiple intertube	Mechanical
X-1......		Pittsburgh	Seam and deep mine	8.70	64.9	22.9	13 868	104	78.7	Horizontal turbulent burners	Electrostatic precipitation
S-13......		75 per cent Fairmount 25 per cent Pa.	...	8.8	63.0	28.2	13 434	...	70	...	...
S-14......		S. Ind.	Strip	8.12	44.23	35.34	11 461	43.3	76 to 85	Injector	Electrostatic precipitation
	MF	MEDIUM (6 TO 11 PER CENT) IGNITION—MORE THAN 88 PER CENT PASSING NO. 325 SIEVE								
J........		Fulton Co. Central Co.	Strip Shaft	9.25 10.40	... 43	... 32	10 500 10 428	... ...	... ...	Crushed coal by cyclone	Electrostatic precipitation
I........		W. Va.	Deep	8	53.0	39.0	13 500	...	75	Foster-Wheeler circular burner	Mechanical and electrostatic precipitation
S-16......		Mo.	Strip	15.3	44.3	40.4	12 400	...	70	...	...
	MC	MEDIUM (6 TO 11 PER CENT) IGNITION—LESS THAN 88 PER CENT PASSING NO. 325 SIEVE								
G........		Central Pa.	Seam	8.21	66.75	21.61	13 365	105	80 to 85	Horizontal turbulent burners	Electrostatic precipitation
K........		Ohio, Ky. W. Va.	... ...	... 8 to 12	... 51 to 59	... 33 to 39	12 820 to 13 930	... 70	50 to 55	Front and tangential	Centrifugal and electrostatic
S-18......		Ohio, E. Ky.	Deep and strip	7.0 to 12.0	51 to 57	34.0	13 500	47 to 55	70	Horizontal pulverizer	Mechanical and electrostatic precipitation
S-19......		E. Ky., W. Va.	Deep	7.0	5.5	34.0	13 300	47 to 55	70	Tangential	Aerotic mechanical
S-20......		Mo.	Strip	15.3	44.3	40.4	12 400	...	70	Tangential and injector	Aerotic precipitation and western precipitation
S-21......		Mo.	Strip	15.3	44.3	40.4	12 400	...	70	Injector	Western precipitation
X-11......		W. Va.	Deep	9.30	56.53	30.01	13 248	...	85	Intervane	Electrostatic precipitation

[Continued on pages 1158 and 1159.]

TABLE I.—DESCRIPTION OF COAL USED TO PRODUCE FLY ASH—*Continued.*

Fly Ash Number	Designation Series	Location of Mine	Type of Mine	Ash, per cent	Fixed Carbon, per cent	Volatile, per cent	Thermal Conductivity Units	Grindability, per cent	Coal Fineness When Burned, Passing No. 200 Sieve, per cent	Type of Firing	Fly Ash Collecting System
	MC	MEDIUM (6 TO 11 PER CENT) IGNITION—LESS THAN 88 PER CENT PASSING NO. 325 SIEVE									
S-23......		Ohio W. Va.	Deep and strip Seam	15.67	...	...	...	...	72	Combustion tangential	...
S-24......		75 per cent Fairmount 25 per cent Pa.	...	8.8	63.0	28.2	13 434	...	70	Tangential	Electrostatic
S-25......		Upper Kittating	Strip	13.47	51.36	35.19	12 652	...	85	Injector	Mechanical
X-10.....		West Pa. West Va.	90 per cent deep 10 per cent strip	8.0	53.0	39.0	13 500	...	75	...	...
	HF	HIGH (711) IGNITION—LESS THAN 88 PER CENT PASSING NO. 325 SIEVE									
L........		[a] Prepared anthracite	Deep mine	13.4	80.6	6.0	12 903	51.5 to 59.4	84	Hony burner	Electrostatic precipitation series with precipitation multicyclone
				33.9	60.6	5.5	9 343	51.9	84	Hony burner	
				9.8	54.5	35.7	13 680	69.1 to 80.6	84	Hony burner	
M........		Prepared anthracite: buckwheat 78 per cent	Deep mine	13.0	80.9	6.1	12 965	51.5 to 59.4	92	Injected vertically downward	Precipitation multicyclone in series with electrostatic precipitation
		silt 22 per cent		33.3	61.7	5.7	9 451	51.9	92	Injected vertically downward	

	HC	HIGH (711) IGNITION—LESS THAN 88 PER CENT PASSING NO. 325 SIEVE									
N........		S.W. Va.	Seam	22.0	60.6	16.0	11 500	...	80	Injector	Mechanical
		[a] Prepared anthracite	Deep mine	12.7	83.4	3.9	12 748	49 to 55	88	Injected vertically downward	Western precipitation multicyclone
				13.4	80.6	6.0	12 903	51.5 to 59.4	84		
S27......		Prepared anthracite	...	33.9	60.6	5.5	9 343	51.9	84	...	...
				9.8	54.5	35.7	13 680	69.1 to 82.6	84	...	...

---

[a] Buckwheat—16.9 per cent; Anthracite silt—9.4 per cent; Bituminous—73.7 per cent.

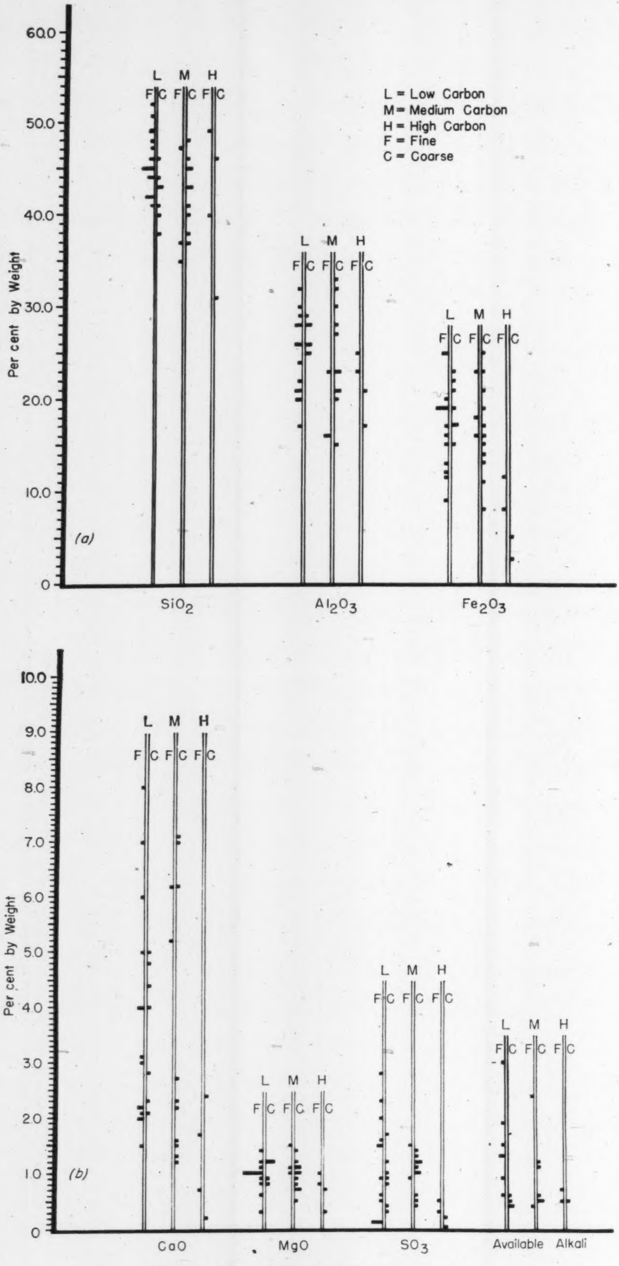


FIG. 1.—Chemical Analysis of Fly Ash.

ples of fly ash as produced in different burning operations, and it is, in fact, difficult to obtain a typical sample of fly ash from a fixed source of supply. The primary ingredient in fly ash is a finely divided, highly siliceous, glassy, spherical component. Residual unburned carbon can virtually always be found in fly ash, together with inclusions of unfused ash. Magnetic and nonmagnetic iron compounds, some alkali, and a limited amount of water-soluble material are also present. Differences between fly ashes usually reflect the extent to which the various ingredients are present and the physical size and form of these components.

The present study was undertaken to establish more precisely the basic structure of fly ash. The results are given in the form of chemical analyses, microscopic studies, X-ray diffraction, and differential thermal analysis. The investigation has been carried out to a large extent on fractional components of the fly ash, since it is felt that this is more revealing than observations made with composite materials.

In addition to the investigation of the fly ash and fractionated samples of fly ash, some information is reported on both unreacted and reacted lime – fly ash mixtures. The latter information is considered to be preliminary in nature, and a more complete report on the reaction compounds of fly ash and lime is expected to be presented at a later date.

In order to make the presentation have wider application, the fly ash samples correspond in large part to material that is being used currently by ASTM committees in round-robin test evaluations. Several of the samples were also used in an earlier series of tests sponsored by subcommittees of ASTM Committees C-1 and C-9 (4). The information in this report may therefore be of some help to ASTM Committees, particularly to Committee C-9 on Concrete and Concrete Aggregates and Committee C-7 on Lime. Conversely, the results obtained in the current round-robin studies will be of assistence in interpreting the information presented in this report.

MATERIALS

Fly Ash:

Table I describes in some detail the sources of fly ash samples used in the study. A questionnaire which was originally sent out by ASTM Committee C-9 in establishing this committee's current round-robin test program is the basis for most of this information. Additional information has been supplied by a number of the companies producing fly ash. It is believed that the samples are representative of the materials available in this country.

Table I includes background information on the type of coal used, the fineness of the coal at the time of burning, the type of firing, and the collection systems used. After making a preliminary chemical analysis of the fly ashes, a group of samples was selected which was considered to represent the complete range of materials. The basis of this selection is virtually identical to that used by ASTM Committees C-9 and C-7 in the abovementioned round-robin test programs.

Figure 1 is a graphical presentation of the analysis of the fly ashes. It will be noted that there are three major groupings consisting of low carbon (0 to 6 per cent), medium carbon (6 to 11 per cent) and high carbon (greater than 11 per cent), designated L, M, and H, respectively. Each of these main groups is then subdivided into two subgroups representing a variation in fineness. The coarse material (designation "C") refers to fly ash which contains less than 88 per cent passing a No. 325 sieve. The fine material (designation "F") contains more than 88 per cent passing a No. 325 sieve. Table II lists the analysis of the fly ashes as rounded averages. The values given for the sample "A" through "N" and those prefaced by the letter "X" are based on results which have been reported by the testing laboratories participating in the ASTM cooperative programs. The remaining analyses are based on values supplied by the fly ash producers.

TABLE II.—FLY ASH ANALYSIS.
(Rounded Values—per cent by weight)

Series	Fly Ash Number	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Ignition Loss	Available Alkali	Retained on No. 325 Mesh	Water-Soluble	Pozzolanic Reactivity (ASTM Specification C 379)
L-F......	A	42	20	25	4.0	0.9	1.5	2.4	0.6	6	2.26	18.38
	S-1	41	21	25	7.0	1.0	0.9	2.0	...	7	...	...
	E	44	20	19	8.0	1.4	2.8	1.3	1.3	5	4.37	19.90
	X-16	48	21	19	5.0	1.2	2.3	1.5	3.0	11	...	...
	S-3	47	28	13	3	1.0	0.6	4.0	...	4	...	...
	S-4	42	28	15	4.0	1.0	1.5	6.0	...	4	...	...
	S-5	45	26	19	1.5	1.0	...	2.4	1.5	11	...	...
	S-6	45	22	20	2.0	0.3	0.1	4.9	0.9	6	...	...
	S-7	44	30	16	2.1	0.8	0.1	3.4	1.3	1	...	...
	S-8	51	26	12	2.0	1.1	0.1	3.2	1.9	2	...	...
	S-9	45	17	17	6.0	1.0	2.0	2.5	...	9	...	...
	S-10	52	29	12	2.2	0.9	0.1	...	...	10	...	...
	S-11	46	24	19	3.1	1.0	1.6	6.0	...	11	...	...
	S-17	49	32	9	2.2	0.6	0.5	4.7	...	2	...	...
L-C......	B	43	26	22	2.1	1.2	0.3	3.3	0.6	28	0.83	11.63
	C	46	26	17	4.0	1.2	0.9	4.0	0.4	13	...	...
	D	41	25	21	4.4	0.8	0.8	5.9	0.4	13	1.50	1.26
	F	43	28	15	4.8	1.2	1.7	5.9	0.5	15	...	...
	X-1	44	29	17	2.8	0.9	0.4	5.0	...	32	...	...
	S-13	38	25	19	5.0	0.8	1.2	5.0	...	32	...	...
	S-14	40	28	23	2.3	...	1.0	...	...	18	...	...
M-F.....	I	37	23	23	6.2	1.1	0.9	6.7	0.4	11	1.63	13.55
	J	35	16	16	5.2	1.0	...	10.1	2.4	3	8.35	22.04
	S-16	47	16	18	...	1.5	1.5	13.0	...	11	...	...
M-C.....	G	40	33	14	2.7	0.8	0.5	7.5	0.5	15	0.88	24.42
	K	41	23	21	1.3	0.9	0.6	9.6	0.5	12	1.81	17.65
	S-18	48	32	8	1.6	0.5	1.0	7.0	1.1	25	...	...
	S-19	46	30	11	1.5	0.7	1.0	6.2	0.6	32	...	...
	S-20	45	15	17	...	1.4	1.4	7.8	...	15	...	...
	S-21	48	21	15	7.0	1.2	1.1	6.3	...	20	...	...
	X-11	43	28	16	2.2	1.1	0.4	7.8	...	24	...	...
	S-23	43	21	25	2.3	1.0	1.3	6.1	...	19	...	...
	S-24	38	27	13	6.2	1.1	1.2	6.6	...	25	...	...
	S-25	45	20	19	1.2	0.7	...	6.9	...	24	...	...
	X-10	37	23	23	7.1	1.0	1.2	6.2	1.2	29	...	...
H-F.....	L	40	23	12	1.7	1.0	0.5	20.0	0.7	11	2.21	13.69
	M	49	25	8	0.7	0.8	0.3	12.5	0.5	10	0.57	25.67
H-C.....	N	46	21	5	2.4	0.7	0.2	22.0	0.5	16	...	22.75
	S-27	31	17	3	0.2	0.3	0.03	40+	...	18	...	...

Lime:

The two types of lime used in the studies represent the same material that is being used by ASTM Committee C-7 in its current round-robin test program. Each of the limes is a blend of several samples, one being a high-calcium lime, the other being a dolomitic lime, type N. The analyses of the limes are given in Table III.

Aggregate:

The sand used in making the test specimens was graded standard Ottawa sand as specified in ASTM Method C 109.[3]

TABLE III.—CHEMICAL ANALYSIS OF LIMES.

	Mono-hydrated Dolomitic Lime, per cent	High Calcium Lime, per cent
Calcium hydroxide	61.8	94.2
Magnesium oxide	33.8	0.6
Calcium carbonate	1.8	2.2
Free water	0.5	1.1
Silicon dioxide	1.4	0.4
Iron and aluminum oxides	0.6	1.2

EXPERIMENTAL PROCEDURES

Sample Designation:

In addition to the designation for the three main series L, M, and H, with subgroups "C" and "F," each fly ash has been individually labeled. The letters "A" through "N" conform to the letters which are currently being used in the ASTM Committees C-7 and C-9 round robin test studies. The samples listed by a number preceded by the letter "X" conform with materials used in previous ASTM studies (**4**). The numbers in this case agree with those used in the earlier investigation. Supplementary samples carry the designation "S." These are listed numerically, ranging from S-1 to S-28.

Sample Preparation:

Representative samples were taken for each of the materials to be tested. The samples were also separated into various fractions using the following techniques:

Sieve Fractions.—A gallon sample of fly ash was passed through a laboratory riffler and a representative 50-g sample was selected for separation by means of dry sieving on a Ro-Tap mechanical sieve. It was found that 15 min sieving time was sufficient to obtain a clean separation.

Magnetic Separation.—Several pounds of the fly ash were treated in a laboratory model of a dry magnetic separator which is capable of removing a clean magnetic fraction. The nonmagnetic residue was also retained for use in the program.[4]

Specific Gravity Fractions.—A blend of carbon tetrachloride and acetylene tetrabromide was prepared and adjusted in proportion to give a specific gravity of 2.00. Carbon tetrabromide and acetylene tetrabromide were used to make a solution of specific gravity of 3.00.

A sample of approximately 25 g of the fly ash was suspended in the solution of specific gravity of 2.00. The suspension was then placed in a centrifuge and the lighter than 2.0 material was removed by decantation and filtration. The heavy fraction was washed in xylene and dried, then placed in the solution of specific gravity 3.0. The centrifuge procedure was repeated giving a second product running between 2.0 and 3.0 in specific gravity and a third product that is greater than 3.0.

Chemical Analyses:

The methods of carrying out the chemical analyses involved the use of applicable portions of ASTM Method of Test

---

[3] Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 58), 1958 Book of ASTM Standards, Part 4, p. 124.

[4] The magnetic separation process has been developed by G. & W. H. Corson, Inc. who have licensed Dings Magnetic Separator Co. to manufacture the machines.

C 311 – 53 T[5] and Specification C 379 – 56 T.[6] A few of the elements, namely, silicon, iron, and sulfur, were determined by the use of an X-ray fluorescent spectrograph using a Norelco machine.

X-Ray Diffraction:

The X-ray studies were performed on two instruments, both Norelco diffractometers with copper X-ray targets operated at 35 kv, one machine having a 90 deg range was operated at 6 ma, the other having a 190 deg range was operated at 18 ma. The latter machine is equipped with a pulse height analysis. The major $\frac{d}{n}$ values were replotted to make comparisons of the samples more convenient.

Differential Thermal Analysis:

Standard techniques were used for differential thermal analyses. The equipment consists of a nichrome-wound tube furnace, a temperature recorder and controllers, and an Inconel block with three thermocouple receptacles approximately $\frac{3}{8}$ in. in diameter and $\frac{3}{8}$ in. deep. These three thermocouple wells are connected in such a manner that one thermocouple is placed in inert alumina for recording the furnace temperature, and the other two thermocouples are connected in a balanced circuit to make it possible to measure differential changes in temperatures in such a way that the reactions taking place in the sample as compared to inert alumina would be suitably recorded. A rate of 10 C per min was used for all of the tests.

Measurements were made on the individual materials as well as on reacted and unreacted lime fly ash.

Preparation of Test Mixtures:

Mixtures of lime and fly ash were prepared as dry mixtures, as wet pastes, and as lime – fly ash – sand mortars. The specimens which were molded and subjected to curing conditions were prepared in accordance with applicable sections of ASTM Specification C 379. The materials reported and proportions of ingredients are as follows:

Mix	Lime	Fly Ash, passing No. 325 Sieve	Proportions, Parts by Weight		
			Lime	Fly Ash	Sand
No. 1....	High calcium	Types D, L, K	1	2	...
No. 2....	Dolomitic	Type D	1	2	...
No. 3....	High calcium	Type D	1	2	10
No. 4....	Dolomitic	Type D	1	2	10

Storage of Test Specimens:

The molded specimens were subjected to four sets of curing conditions as follows:

1. Ambient condition—partially exposed area on roof of building. Aging of specimen to be of several years duration.
2. 95 to 100 per cent relative humidity at 73 ± 3 F. Aging of specimen to be 1 yr.
3. Sealed containers at 140 F. Aging of specimen 3, 7 and 14 days.
4. High-pressure autoclave 300-lb pressure for 3 hr.

The specimens subjected to X-ray analysis after curing were pulverized prior to making measurements.

TEST RESULTS

Chemical Analyses:

The results of the chemical analyses are presented in Tables II and IV and in Figs. 1 and 2. Figure 1 is used to assist the reader in comparing the effect of ignition loss and fineness on the other chem-

---

[5] Tentative Methods of Test for Fly Ash as an Admixture for Portland Cement Concrete (C 311 – 53 T), 1953 Supplement to Book of ASTM Standards, Part 3, p. 253.

[6] Tentative Specification for Fly Ash for Use as a Pozzolanic Material with Lime (C 379 – 56 T), 1958 Book of ASTM Standards, Part 4, p. 229.

TABLE IV.—ANALYSIS OF FLY ASH AND FLY ASH FRACTIONS.

Fly Ash Designation	Constituents	Rounded Values of Table II	Composite of Sieve Fractions	Fraction Retained on No. 100, per cent	Fraction Passing No. 100, Retained on No. 200, per cent	Fraction Passing No. 200, Retained on No. 325, per cent	Fraction Passing No. 325 per cent	Magnetic Fraction	Nonmagnetic Fraction	Specific Gravity		
										<2.0	2.0 to 3.0	>3.0
“A”	SiO_2	42.00	40.00	31.60	33.00	36.70	40.40	10.10	47.00	51.74	41.40	15.28
LF	Al_2O_3	20.00	19.00	17.50	14.25	16.10	18.75	7.10	21.70	27.05	32.20	13.10
	Fe_2O_3	25.00	27.50	12.60	17.90	28.10	28.05	79.20	14.30	8.45	10.15	60.30
	CaO	4.00	5.00	4.20	7.13	6.93	4.94	0.78	5.20	0.82	5.05	5.60
	MgO	0.90	1.40	2.00	1.88	1.92	1.30	3.00	2.00	2.63	1.03	3.00
	SO_3	1.50	1.55	1.80	0.55	2.00	1.47	0.73	1.28	0.48	0.77	1.90
	Ignition Loss	2.40	4.70	31.20	25.20	6.90	2.63	0.30	5.00	5.74	7.68	0.70
	^{a}Yield	...	...	1.54	3.90	7.88	86.66	18.00	82.00	...	...	...
“D”	SiO_2	41.00	39.00	32.70	38.50	35.90	40.50	13.10	44.40	41.90	42.42	18.50
LC	Al_2O_3	25.00	25.50	21.00	21.30	20.50	25.60	9.70	28.50	26.80	25.46	16.10
	Fe_2O_3	21.00	23.60	19.50	24.60	30.30	24.80	71.10	13.50	7.04	12.04	58.00
	CaO	4.40	4.40	4.00	2.80	3.80	4.50	1.20	5.00	1.32	3.38	2.48
	MgO	0.80	2.10	2.20	2.30	0.98	2.20	3.20	2.50	1.20	0.78	3.00
	SO_3	0.80	1.00	0.95	0.60	1.70	0.60	0.63	0.67	0.45	0.54	0.69
	Ignition Loss	5.9	3.40	19.30	5.50	6.60	2.30	0.03	3.50	18.84	9.39	0.68
	^{a}Yield	...	...	2.74	4.12	11.10	81.92	18.00	82.00	...	...	...
“I”	SiO_2	37.00	36.50	20.80	28.50	30.92	38.02	7.40	41.70	39.10	43.18	10.60
MF	Al_2O_3	23.00	22.00	15.20	19.15	13.70	23.05	3.60	26.50	29.67	26.70	18.10
	Fe_2O_3	23.00	25.00	9.20	13.30	32.10	25.10	83.20	13.70	6.03	8.10	56.40
	CaO	6.20	6.20	4.20	4.13	5.04	6.45	0.90	7.60	1.97	5.78	9.08
	MgO	1.10	2.20	2.10	2.72	1.72	2.33	3.00	2.30	2.92	2.52	1.65
	SO_3	0.90	1.10	2.00	2.63	2.45	0.88	0.62	0.85	0.46	0.57	1.08
	Ignition Loss	6.70	6.50	47.80	29.30	13.70	3.97	0.42	7.00	16.92	11.45	1.42
	^{a}Yield	...	...	1.44	2.36	12.88	83.22	10.00	90.00	...	...	...
“J”	SiO_2	35.00	36.30	31.80	31.67	32.66	39.02	14.80	38.50	39.26	34.78	12.00
MC	Al_2O_3	16.00	21.20	18.00	18.75	19.50	21.60	7.20	20.60	29.44	18.75	11.80
	Fe_2O_3	16.00	16.00	15.20	14.40	17.50	15.40	67.30	15.80	7.96	14.05	68.00
	CaO	5.20	5.40	3.20	5.44	7.00	4.72	2.40	4.00	2.47	4.55	1.65
	MgO	1.00	2.80	1.25	1.81	2.18	3.47	2.00	3.10	3.84	4.90	1.36
	SO_3	...	4.50	4.10	6.10	5.45	5.00	2.90	...	0.55	1.95	4.05
	Ignition Loss	10.00	12.00	24.80	17.70	15.67	8.50	4.50	13.00	16.40	20.72	0.74
	^{a}Yield	...	...	4.36	5.10	32.70	57.84	4.30	95.70	...	...	...
“K”	SiO_2	41.00	39.00	24.10	33.74	36.82	39.34	13.80	44.40	39.80	45.00	8.00
MC	Al_2O_3	23.00	23.00	11.75	16.80	17.55	25.45	7.90	24.50	24.01	24.95	16.00
	Fe_2O_3	21.00	22.00	9.00	10.55	23.15	22.20	74.30	12.00	7.14	11.10	66.80
	CaO	1.30	2.10	0.25	0.97	1.65	2.31	0.70	0.70	0.50	2.06	1.15
	MgO	0.9	1.70	3.50	1.98	2.46	1.43	2.10	1.50	1.59	1.76	1.82
	SO_3	0.6	0.80	1.20	2.95	1.62	0.52	0.49	0.73	0.69	0.44	0.41
	Ignition Loss	9.6	9.2	50.00	32.55	14.53	7.50	0.24	12.50	22.30	13.45	3.37
	^{a}Yield	...	...	1.40	2.48	21.30	74.80	13.10	86.90	...	...	...
“X-10”	SiO_2	37.00	37.00	32.00	35.50	33.50	38.30	10.64	46.66	46.60	45.00	22.50
MC	Al_2O_3	23.00	24.00	14.70	18.00	18.80	26.30	8.20	29.30	30.30	30.10	23.90
	Fe_2O_3	23.00	25.00	16.80	36.20	32.60	21.20	71.80	10.00	7.70	9.50	42.30
	CaO	7.10	6.00	2.80	2.80	4.25	5.40	1.23	6.50	2.80	5.00	5.80
	MgO	1.00	2.00	1.20	0.70	2.00	2.20	1.69	2.70	0.90	2.20	1.50
	SO_3	1.20	1.10	1.00	0.55	1.84	0.98	0.17	1.00	0.57	0.72	1.22
	Ignition Loss	6.20	5.00	26.60	2.20	5.10	3.10	4.0	2.92	9.70	6.50	0.40
	^{a}Yield	...	...	5.70	10.90	17.36	66.90	22.80	77.20	...	...	...
“L”	SiO_2	40.00	38.00	21.60	23.90	27.76	40.30	11.20	39.60	18.90	49.22	26.26
HF	Al_2O_3	23.00	22.40	16.00	8.10	12.05	24.85	7.30	25.10	13.60	26.75	26.80
	Fe_2O_3	12.00	11.30	3.50	4.90	7.15	12.40	79.20	7.00	3.00	8.55	41.40
	CaO	1.70	1.90	0.30	0.41	0.82	2.15	0.80	0.95	0.66	2.31	1.65
	MgO	1.00	1.60	2.50	2.17	1.74	1.52	1.80	2.70	2.19	1.52	3.20
	SO_3	0.50	0.50	1.20	0.70	0.55	0.52	0.80	0.62	0.72	0.46	0.39
	Ignition Loss	20.00	22.80	54.50	58.70	47.92	15.75	0.50	23.50	60.78	8.17	3.12
	^{a}Yield	...	...	1.44	4.36	12.70	81.50	4.20	95.80	...	...	...

[Continued on page 1166.]

TABLE IV.—ANALYSIS OF FLY ASH AND FLY ASH FRACTIONS—*Concluded.*

Fly Ash Designation	Constituents	Rounded Values of Table II	Composite of Sieve Fractions	Fraction Retained on No. 100 per cent	Fraction Passing No. 100, Retained on No. 200, per cent	Fraction Passing No. 200, Retained on No. 325, per cent	Fraction Passing No. 325, per cent	Magnetic Fraction	Nonmagnetic Fraction	Specific Gavity		
										<2.0	2.0 to 3.0	>3.0
"N"	SiO_2	46.00	45.00	37.80	41.80	46.70	45.60	20.20	49.00	42.36	56.84	30.30
HC	Al_2O_3	21.00	22.00	16.50	21.25	16.81	23.95	12.60	22.40	25.30	18.70	17.40
	Fe_2O_3	5.00	6.00	6.70	4.75	7.59	5.65	58.40	4.60	4.15	6.25	38.20
	CaO	2.40	3.00	0.96	0.99	1.31	6.26	0.40	2.20	0.66	3.05	3.55
	MgO	0.70	1.60	1.60	0.73	1.96	1.52	1.95	2.80	1.29	1.73	2.60
	SO_3	0.20	0.14	0.45	0.70	0.44	0.17	0.37	0.19	0.24	0.21	0.37
	Ignition Loss	2.20	19.50	34.00	25.28	20.70	16.70	4.20	16.20	21.58	8.40	2.86
	[a]Yield	...	...	2.20	7.00	19.20	71.60	2.50	97.50	...	...	...

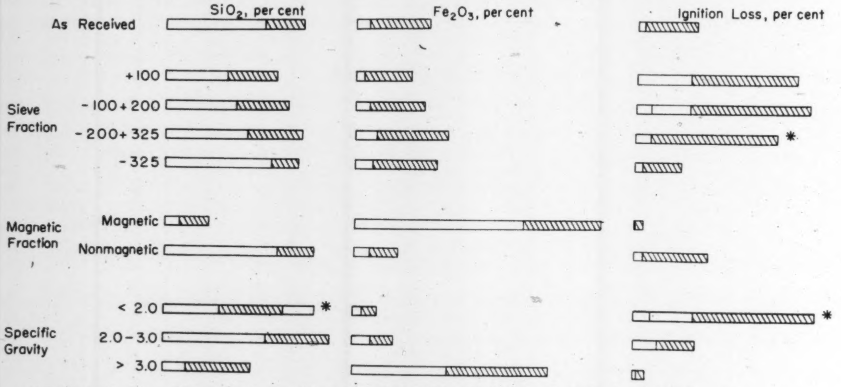
[a] The yield is given in weight percentage of the composite fly ash.

ical constituents for the different fly ashes. Table IV is of particular importance since it shows the considerable variation to be found in the various fractions of fly ash. The fly ash samples used in preparing the various fractions showed some deviation in analysis between the individual composite samples and the averages obtained in the round-robin tests. Both sets of values are therefore given in Table IV.

The analyses given for the magnetic fractions are in close agreement with the products produced commercially. The improvement in quality that can be realized by removal of the coarse fractions is also of interest. The coarse fraction is considerably higher in carbon content; the fine fraction shows higher silica and alumina. A graphical analysis of some of the results listed in Table IV is presented in Fig. 2. The shaded section of the bars gives the significant range of values for each constituent (maximum and minimum values).

Microscopic Examination:

The examination of fly ash by means of a petrographic microscope has not been found to be too fruitful insofar as the identification of the presence of crystalline fractions is concerned. The bulk of the material is found to be glassy in na-

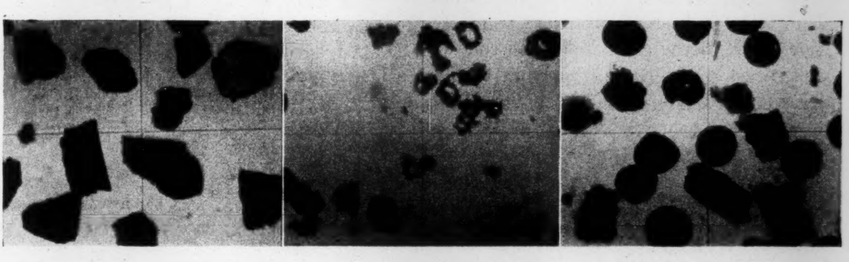


* Isolated value shown not considered sufficiently reliable to include in shaded area.

FIG. 2.—Analysis of Fly Ash and Fly Ash Fractions.

Passing No. 100, retained on No. 200 mesh sieve

Fly ash L Fly ash K Fly ash D



Passing No. 200, retained on No. 325 mesh sieve

Fly ash L Fly ash K Fly ash D

Passing No. 325 mesh sieve

Fly ash L Fly ash K Fly ash D

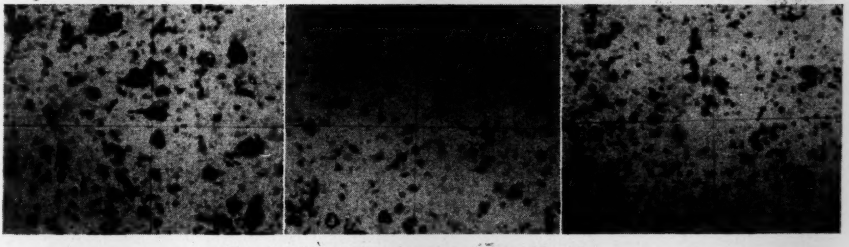


FIG. 3.—Microphotographs of Sieve Fractions.

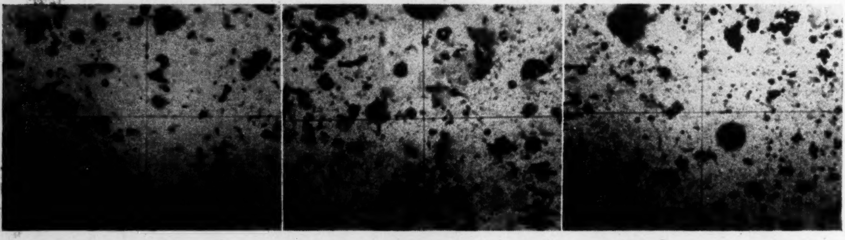
Fly ash L contains a large percentage of angular, black particles, probably unburned or partially burned coal. This contaminant is prominent in the fractions above No. 325 and appears, although in a lesser degree, in the minus No. 325 mesh material.

Fly ash K contains the rounded, open structured black cinder fragments usually associated with the coarse sieve fraction of fly ash. Fly ash K exhibits this material to an unusual degree, although the minus No. 325 fraction is relatively clear of the cinder.

Fly ash D is a well-formed ash, exhibiting typical multicolored spheroidal glass shapes even in the coarse fraction.

NOTE: Very little difference can be observed between fly ashes D and K in the minus No. 325 fraction. Fly ash L would probably lose the remaining angular particles if a No. 400 mesh sieve were used, in which case it, too, would resemble the other ashes.

ture, the crystals being far too small to lend themselves to easy identification. Efforts to make measurements of refractive index of the glass indicated that fly ash shows a very broad range of values. This is obviously the result of variations in the percentages of alumina and iron in the siliceous component. Some compound formation has been indicated in reacted "lime - fly ash - slag composition" reported previously (5). Attempts to study fly ash with the electron microscope have yielded little information to date. Figures 3 to 5 illustrate the type of results that have been obtained using low-power objectives and reflected illumination.


Nonmagnetic

Fly ash L Fly ash K Fly ash D

Magnetic

Fly ash L Fly ash K Fly ash D

FIG. 4.—Micrographs of Magnetic Separation Fractions.

In general, the nonmagnetic fractions follow the same pattern as the screened fractions. Fly ash L shows the prominent angular shapes; fly ash K the cinder and fly ash D the typical spheres. The magnetic fractions of these three ashes are, however, indistinguishable from each other. The "chain" structure is typical of this magnetic material—being caused by mutual attraction of residual magnetic poles induced from the magnetic separator. Color of the particles ranges from a dark brown to black.

X-Ray Diffraction:

The data are given in graphical form using conventional means of plotting the X-ray spectra in terms of interplanar spacing, represented as $\frac{d}{n}$ values. These values are derived from the fundamental Bragg equation $n\lambda = 2d \sin \theta$ where n is any positive integer, λ is wavelength in Angstrom units, d is interplanar distance in Angströms and θ is the angle of diffraction in degrees. The lines given are the most prominent and are easily identified. Four basic series of comparisons have been made as follows:

1. Comparison of different fly ash samples with known minerals.
2. Comparison of fractions of fly ash

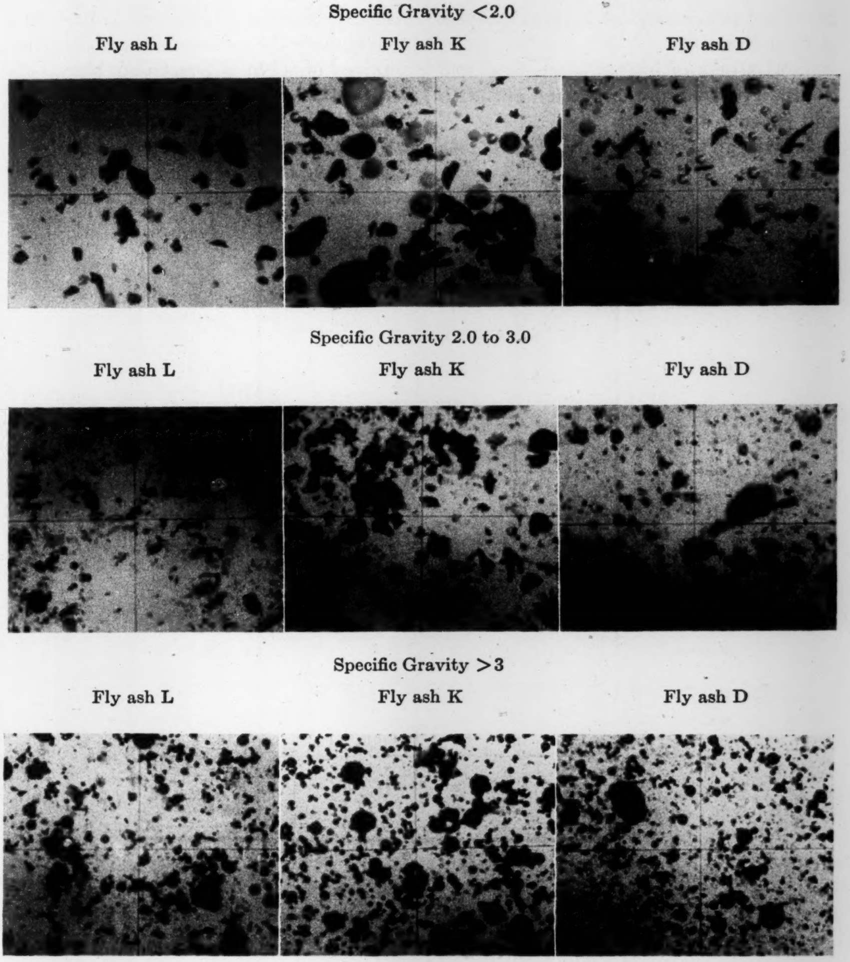


FIG. 5.—Micrographs of Specific Gravity Separation Fractions.

Practically all of the angular shaped particles of fly ash L appear in the <2.0 fraction. Since few coals range higher than 1.7 in specific gravity, a means of recovery with improvement in the resulting coal-free ash is suggested. The only other material appearing in the <2.0 fraction of this fly ash is a milk-white glass in a form resembling miniature snowballs.

A concentration of the open structured cinder fragments appears in the <2.0 fraction of fly ash K and (to a lesser extent) in fly ash D. The milk-white glass, some lightly tinted glass and (particularly in fly ash D) some transparent glass spheres make up the remaining material in these fractions. The separation of the cinder fragments is by no means as complete as the angular fragments were in fly ash L since much of the cinder also appears in the 2.0–3.0 fraction.

The 2.0–3.0 fractions of the three fly ashes are more or less indistinguishable from each other, although fly ash L now appears more free of carbonaceous material due to the excellent separation of the angular particles. The heavy fraction (>3.0) of fly ash K closely resembles a magnetic fraction. The material is highly colored with very little ultra fine material present. Surprisingly enough, the L and D fly ash samples of this fraction contained, along with this highly colored material, a large amount of clear spheres of very small diameter. Whether this is a very heavy glass or whether difficulties in making the separation account for the presence of this material remains a question.

described previously for several samples of fly ash.

3. Mixtures of high-calcium lime and fly ash—reacted and unreacted and with and without aggregate.

4. Mixtures of dolomitic lime-fly ash mixtures.

The curing conditions covered in this report include the 140 C accelerated cure and the high-pressure autoclave cure. The ambient temperature results are not reported inasmuch as the extended curing time required did not permit their inclusion in this report. Most of these samples will become available late 1959 the remaining early in 1960.

Comparison of Fly Ash Samples.—Figure 6 shows line diagrams for four fly ashes. These materials represent samples of varying fineness and varying carbon content. Samples D, K, and L have approximately the same amount of material retained on a No. 325 sieve but show carbon variation from 5.9 to 20 per cent. The sample X-10 has a carbon content of 6.2 per cent but a residue on a No. 325 mesh sieve of 29 per cent. Fly ash X-10 was also fired to 1000 C for ½ hr in an oxidizing atmosphere. The purpose of this was to assist in studying the type of iron minerals which may be present and to determine if any further information could be obtained from new compounds developed by firing. For example, it is possible that devitrification could be produced by the firing procedure used.

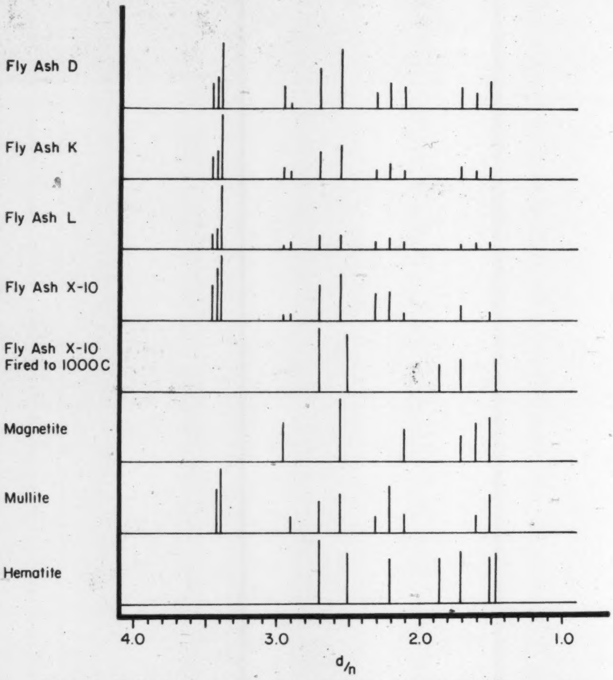


FIG. 6.—Line Diagrams for Four Fly Ashes and Three Minerals.

In addition to the fly ash samples, line diagrams are included for the minerals magnetite, hematite, and mullite. In general, it can be stated that the major

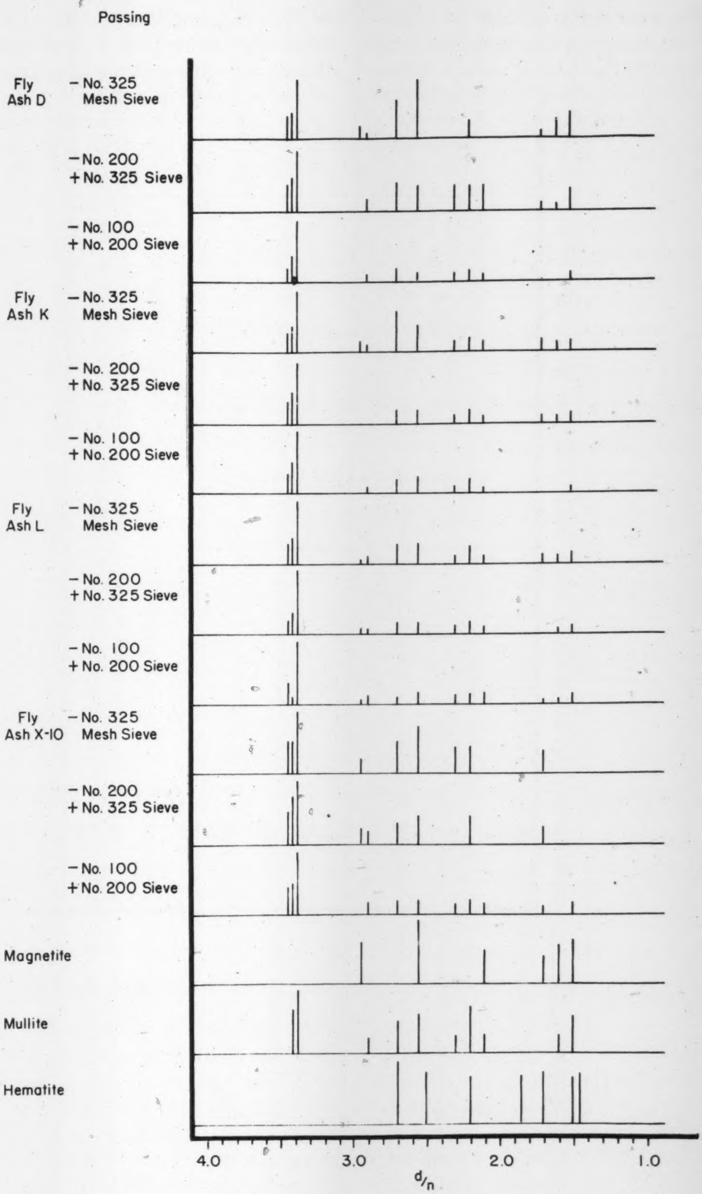


FIG. 7.—Line Diagrams Showing Sieve Fractions for Four Samples of Fly Ash and Three Minerals.

crystalline material in fly ash is magnetite with some indication of the presence of mullite. In the glassy matrix which comprises the bulk of the fly ash, other crystalline materials, probably devitrification products, are detectable, but because of the large amount of glass and the small crystalline size, poor resolution is obtained in the patterns. In the example X-10 which was fired to 1000 C there is an indication that the magnetite has been converted to hematite. Of interest in comparing the fly ash samples (though not shown in the diagram), is an amorphous hump that seems to be indicative of the carbon content of the fly ash. This amorphous hump covers the range of 3.0 Å to 4.9 Å and is more prevalent in the samples higher in carbon content.

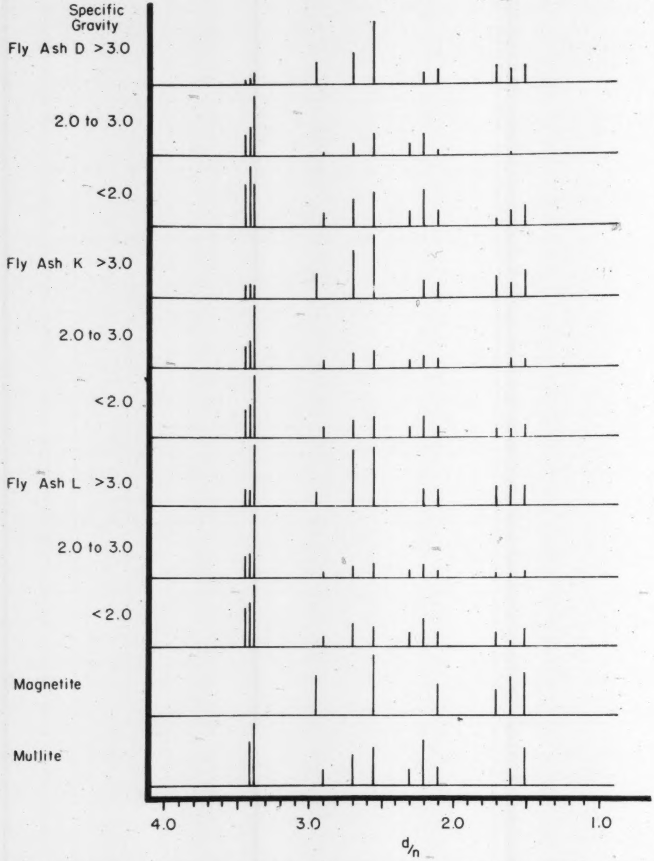


FIG. 8.—Line Diagrams for Three Fly Ash Samples Separated into Different Specific Gravity Fractions.

Comparison of Fly Ash Fractions.—Figure 7 shows the line diagrams for the sieve fractions as obtained from the four samples of fly ash, D, K, L, and X-10. The samples show the presence of magnetite and also the dominant 3.36 Å line with differences in intensity being developed as the material is separated into fractions. For instance, the 2.51 Å line in sample X-10 is definitely stronger in the fine material than it is in the coarse material. It is also significant that the amorphous bulge caused by the carbon fraction is found in the coarse fractions and is of lesser prominence in the material passing the No. 325 sieve.

Figure 8 shows the comparisons for three fly ash samples that have been separated into different specific gravity fractions. The high specific gravity shows more prominent magnetite peaks. It will be noted that the 3.36 Å peak is absent in the heavy fractions. The 1.53 Å peak occurs in the lightest fraction. There is little or no evidence of the carbon bulge in the material greater than specific gravity 3.0.

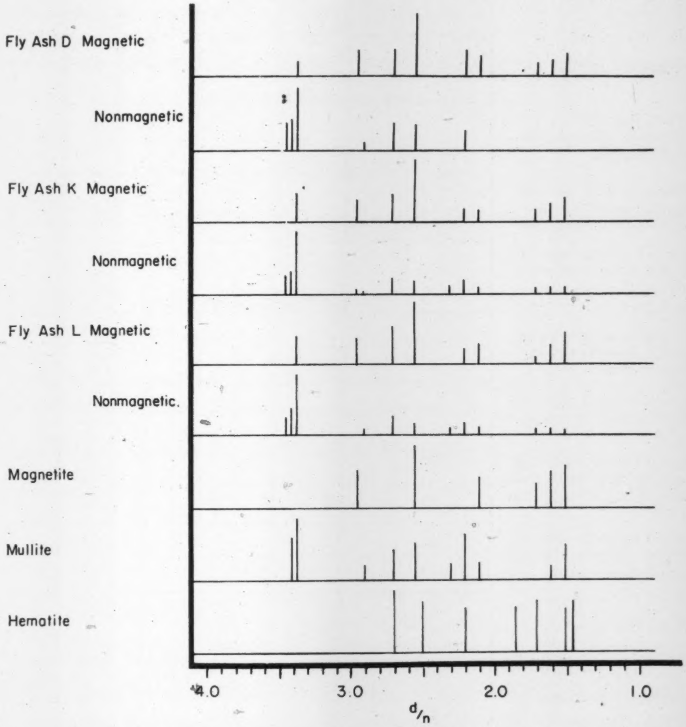


FIG. 9.—Line Diagrams Showing Differences Between Magnetic and Nonmagnetic Fractions for Three Fly Ashes and Three Minerals.

Figure 9 shows the difference between magnetic and nonmagnetic fractions for three of the fly ashes. It is evident in each case that the magnetic fraction is rich in magnetite. The nonmagnetic fractions show the presence of minor amounts of magnetite but contain the most of those peaks corresponding to the major mullite peak. However, the 1.53 Å peak is absent in fly ash D.

High-Calcium Lime – Fly Ash Mixtures.—Figure 10 gives the line diagrams for a group of mixtures that were made from high-calcium lime and fly ash, sample D. The patterns are given for the unreacted mixtures and for the reacted mixtures under several conditions of curing. It will be noted that the mixing of lime and fly ash results in a pattern which contains the prominent lines of each ingredient. It is also to be noted that as the curing condition becomes either more severe, such as in the case of the autoclave sample, or more prolonged, as shown in the 140 C cures, the prominent calcium hydroxide limes are reduced in intensity. Of particular interest is the development of a composite peak in the area of approximately 3.05 Å and the indicated new lines at 2.77, 2.97, and 3.31 Å. None of these lines is particularly sharp. No attempt has been made to

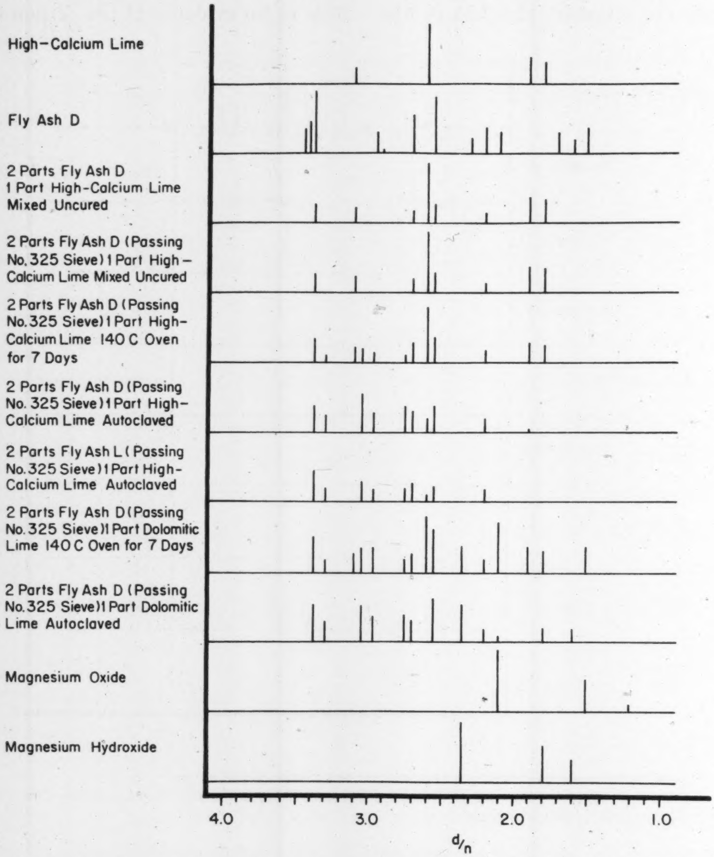


FIG. 10.—Line Diagrams for Mixtures Made from High-Calcium Lime and Fly Ash Samples D and L.

identify these peaks as yet, although there is indication that they fall in the range of a number of hydrated calcium silicates.

Dolomitic Lime – Fly Ash Mixtures.—The X-ray studies made with dolomitic lime appear to give similar results to those obtained with high-calcium lime with minor variations. For instance, the composite peaks observed at approximately 3.05 Å are shaped differently with the dolomitic lime although it is equally prominent. Figure 10 includes a few of the diagrams obtained with dolomitic lime. It will be noted that the magnesium oxide peaks have nearly disappeared in the autoclave samples and have been replaced in part with magnesium hydroxide. Figure 10 also includes a pattern obtained with a different sample of fly ash to indicate the similarity of results that have been found in this work.

Differential Thermal Analysis (DTA):

Fly ash with differing carbon contents, beneficiated fly ash and lime – fly ash mixtures, when run with normal differential thermal analysis (DTA) procedures, show an exothermic reaction which reaches a peak at about 600 C (Fig. 11(*d*)). Since it is noted that the lower carbon ashes showed reduced reactions, it is felt that this is probably the carbon oxidation. The erratic results obtained at temperatures of 900 C and above are readily understood when the samples are examined at the end of the run. Since the temperatures reached are in the solidifying range for fly ash, the samples have the appearance of a fired brick. Considerable damage to the thermocouple required a replacement for each high-temperature run.

A magnetic fraction of fly ash having an abnormally low carbon content (Fig. 11(*c*)) (0.08 per cent ignition loss) was reacted with high-calcium lime by autoclaving at 300 C for 3 hr. The DTA curves for the reacted and unreacted mixtures are shown in Figs. 11(*a*) and (*b*). The chief difference noted is the marked decrease in the 500 C endo-

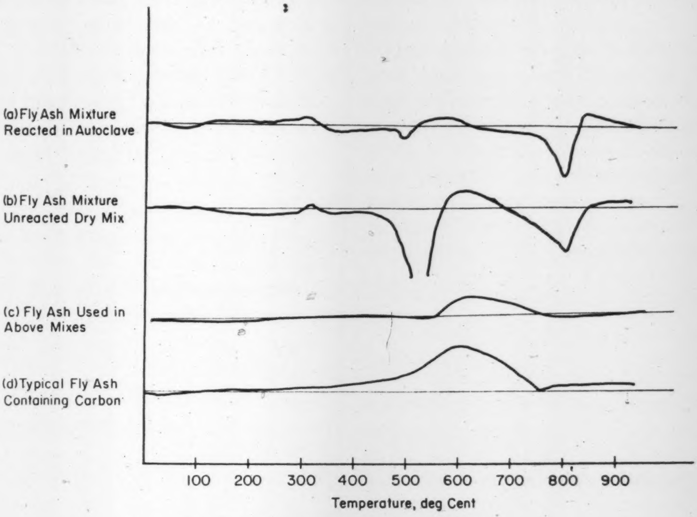


FIG. 11.—Fly Ash – Lime DTA Curves.

thermic valley for the reacted sample which is undoubtedly due to the disappearance of $Ca(OH)_2$. The endothermic reaction present in both samples at 800 C may be due to the decomposition of $CaCO_3$ formed with the CO_2 developed during the carbon oxidation. The small rounded exothermic peak at 850 C in the autoclaved sample is noteworthy, since it is similar to the calcium silicate peaks observed by Kalousek (6). The complete identification and nature of these reactions is being further studied. It should be noted that with relatively simple portland-cement mixtures, considerable difficulty is encountered in positive identification of DTA peaks. With complex lime – fly ash mixtures, this may prove virtually impossible. However, it is felt that if the results are correlated with strength or X-ray data, DTA may prove a useful tool for initial evaluations.

SUMMARY

A group of fly ash samples, representative of material available in this country, has been studied by means of optical microscope, X-ray diffraction and differential thermal analysis. It is indicated that considerable variation exists between samples and that the make-up of fly ash is heterogeneous and complex. Microscopic examination points to the presence of unfused, porous and clinker-like material in the low specific gravity and in the coarse fractions. It is suggested that removal of such contaminants by beneficiation can give more consistent material with improved siliceous glass or pozzolan component.

X-ray diffraction tests show the presence of some crystalline material, magnetite, and possibly mullite. The separation of the fly ash into fractions based on sieve size, specific gravity and magnetic fraction has been found to have a substantial effect on the location of the crystalline material. The unburned carbon is also found to concentrate in some of the separate fractions. Reactions of fly ash fractions with lime have been studied and found to give some indication of compound formation tentatively located in the X-ray region occupied by hydrated calcium silicates and aluminum silicates. Autoclaving resulted in a substantial removal of the $Ca(OH)_2$ and MgO peaks.

DTA studies support the findings of the X-ray studies to a large extent. Of interest is the development of a small rounded exothermic peak at 850 C in the autoclaved sample of lime and magnetic fly ash fraction. The development of strong pozzolanic properties has been recognized in the magnetic fractions produced commercially. The DTA curves point to the possible formation of silicates which may be equivalent to those indicated in the X-ray diffraction studies.

Acknowledgment:

The author greatly appreciates the valuable assistance he has received, especially from William Bower of Rutgers University, Cyril Presgrave of G. & W. H. Corson, Inc., and to many others of the staff of the Corson Laboratory who have contributed their efforts to this report.

REFERENCES

(1) Literature Surveys:
- (*a*) R. E. Morgan, "Utilization of Fly Ash," *Information Circular No. 7635*, Bureau of Mines (1952).
- (*b*) C. E. Littlejohn, "A Literature Review of the Utilization of Fly Ash," *Engineering Experiment Bulletin*, Clemson A & M College, No. 6 (1954).

(2) (*a*) "Laboratory Report," *No. Pet-121*, U. S. Dept. of Interior, Bureau of Reclamation (1957). (Bibliography attached).

(*b*) R. H. Brink and W. J. Halstead, "Studies Relating to the Testing of Fly Ash in Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 1161 (1956).

(3) W. A. Selvig and F. H. Gibson, *Bulletin No. 567*, Bureau of Mines (1956).

(4) L. J. Minnick, "Investigations Relating to the Use of Fly Ash as a Pozzolanic Material and as an Admixture in Portland Cement Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 1129 (1954).

(5) L. J. Minnick, "Lime Fly Ash Soil Compositions in Highways," *Proceedings*, Highway Research Board, Vol. 31, p. 527 (1952).

(6) G. L. Kalousek, "The Reactions of Cement Hydration at Elevated Temperatures," *Proceedings*, 3rd International Symposium of the Chemistry of Cement London, England, Cement and Concrete Association, London, p. 334 (1952).

EFFECTIVENESS OF MINERAL ADMIXTURES IN PREVENTING EXCESSIVE EXPANSION OF CONCRETE DUE TO ALKALI-AGGREGATE REACTION*†

By Leonard Pepper and Bryant Mather[1]

Synopsis

Twenty materials, representing eight different classes of mineral admixtures, were evaluated, using both chemical and mortar-bar test methods, for their effectiveness in preventing excessive expansion of concrete due to alkali-aggregate reaction. It was found that the chemical tests cannot be used with reliance to evaluate effectiveness. Each of the replacement materials evaluated will prevent excessive expansion if a sufficient quantity is used. Correlations were found between effectiveness and: fineness, dissolved silica, and percentage of alkali retained by reaction product.

Five of the materials tested (a fly ash, a tuff, a calcined shale, a calcined diatomite, and an uncalcined diatomite) showed a reduction in alkalinity of 40 per cent or more when tested by the quick chemical test. All of these except the fly ash met the requirement proposed by Moran and Gilliland for the relationship between reduction in alkalinity and silica solubility.

Six of the materials tested (two slags, a fly ash, a pumicite, and two calcined shales) reduced mortar-bar expansion at least 75 per cent with high-alkali cement and Pyrex glass aggregates when used as 50, 45, 35, and 30 per cent replacements of the cement.

Calculations were made that suggest that the minimum quantity of each material required for effective prevention of excessive expansion ranged from 10 per cent for the synthetic silica glass to 45 per cent for one of the slags. By groups, these calculated minimum percentages were: calcined shales, 19 to 29; uncalcined diatomite, 22; volcanic glasses, 32 to 36; slags, 39 to 45; and fly ashes, 40 to 44.

The investigation of mineral admixtures as cement-replacement materials was initiated by the Office of the Chief of Engineers in 1950 as part of the Civil Works Investigations Program with the purpose of ascertaining the degree to which portland cement may be advantageously replaced by other materials, considering cost and the quality of the resulting concrete. This paper deals with that part of the investigation that was concerned with the ability of these materials to prevent excessive expansion of concrete due to alkali-aggregate reaction.

Many tests to determine pozzolanic activity of mineral admixtures have

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

† This paper is based on Technical Report No 6-481, U. S. Army Engineer Waterways Experiment Station, July, 1958.

[1] Chemist, Chief of Chemistry Section, and Civil Engineer, Chief of Special Investigation Branch, respectively, U. S. Army Engineer Waterways Experiment Station, Concrete Division, Jackson, Miss.

TABLE I.—RESULTS OF TESTS OF PORTLAND CEMENTS.

Test Result	Type I Cement		Type II Cement
	High-alkali	Low-alkali	Medium-alkali
CHEMICAL DATA			
Major components:			
SiO_2, per cent	20.6	20.1	23.1
Al_2O_3, per cent	5.4	6.6	5.0
Fe_2O_3, per cent	3.9	3.1	3.9
CaO, per cent	61.0	65.5	61.1
MgO, per cent	3.9	1.2	2.8
SO_3, per cent	2.1	2.0	2.0
Loss on ignition, per cent	1.9	0.97	1.6
Minor components:			
Na_2O, per cent	0.62	0.11	0.23
K_2O, per cent	0.68	0.39	0.69
Total as Na_2O, per cent	1.07	0.37	0.68
P_2O_5, per cent	0.13	0.20	...
Mn_2O_3, per cent	0.07	0.12	...
Separate determinations:			
Insoluble residue, per cent	0.54	0.12	0.23
Chloroform solubility, per cent	0.002	0.004	...
Moisture content, per cent	0.36	0.19	...
Calculated compounds:			
C_3S, per cent	44	56	29
C_2S, per cent	26	16	44
C_3A, per cent	8	12	7
C_4AF, per cent	12	9	12
$CaSO_4$, per cent	4	3	3
PHYSICAL DATA			
Fineness, passing No. 325 sieve, per cent	90.1	93.2	94.8
Fineness, Blaine, sq cm per g	3765	3450	3590
Specific gravity	3.11	3.15	3.17
Time of setting (Gillmore):			
Initial, hr:min	2:50	2:45	5:30
Final, hr:min	4:20	5:45	8:00
Autoclave expansion, per cent	0.28	0.14	0.10
Normal consistency, per cent	24.0	27.2	24.6
Air in mortar, per cent	10.2	7.6	8.8
Compressive strength, psi:			
3 days	1510	2600	1745
7 days	2065	3900	2665
28 days	3065	5565	5140
Heat of hydration, cal per g (70 F water-cement ratio = 0.40):			
3 days	56	66	...
7 days	65	84	71
28 days	71	99	82
6 months	86	106	...
1 yr	86	111	89
Bleeding (water-cement ratio = 0.4):			
Rate, ml per sq cm per sec × 10^6	64	59	37
Capacity, ml per cc × 10^3	18	15	7
Bleeding (water-cement ratio = 0.45):			
Rate	114	...	37
Capacity	29	...	7

TABLE II.—RESULTS OF TESTS OF REPLACEMENT MATERIALS AND HYDRATED LIME.

Test Result	Granulated Water-quenched Iron Blast-furnace Slag		Natural Cement		Fly Ash				Natural Volcanic Glasses				Calcined Shales			Unc D AD-15	Uncalcined Quartz			Syn Silica Glass AD-34	Hydrated Lime AD-31
									Pumicite		Tuff AD-11	Obs AD-12									
	I RC-198	II RC-216 (B)	I RC-214	II RC-215	I AD-3	II AD-7	III AD-8	IV AD-9	(F) AD-6	(L) AD-10			C Sh M AD-5	C Sh N AD-13	Cal D AD-14		SiO₂ Flour AD-35	Air Float AD-36	Ad-Mix AD-37		
CHEMICAL DATA																					
Component:																					
SiO_2, per cent	38.8	37.9	21.0	24.9	47.2	47.4	38.2	44.9	68.8	68.1	69.8	74.8	61.2	70.1	71.1	76.7	99.8	91.4	95.3	98.6	1.03
Al_2O_3, per cent	11.3	13.3	5.2	5.7	19.5	34.0	25.7	34.0	14.8	15.8	14.9	13.8	12.5	19.3	14.9	12.0	0.16	4.8	2.6	0.0	0.93
Fe_2O_3, per cent	0.52	1.0	2.1	4.1	18.2	9.0	16.3	6.5	1.4	1.9	0.85	1.2	4.5	5.9	4.1	2.2	0.05	1.3	0.09	0.0	0.15
CaO, per cent	43.6	35.8	47.7	46.6	5.3	2.3	3.9	2.3	0.65	1.9	1.4	0.27	8.6	0.22	1.02	0.60	0.10	0.42	0.0	0.0	72.7
MgO, per cent	2.0	10.2	15.3	5.8	1.2	1.2	0.9	0.4	0.33	0.97	0.00	0.00	3.0	0.85	2.3	0.70	0.0	0.09	0.0	0.0	1.1
SO_3, per cent	0.08	0.07	2.1	1.7	2.2	0.7	0.60	0.34	0.03	0.02	0.00	0.00	0.44	0.10	1.33	0.05	0.01	0.0	0.0	0.0	0.05
Loss on ignition, per cent	1.35[a]	0.56[a]	6.2	11.2	0.8	3.9	12.2	7.9	3.9	1.99	6.6	0.69	6.8	1.26	2.5	3.2	0.13	1.8	0.74	1.2	24.3
Na_2O, flame, per cent	0.20	0.24	0.11	0.26	1.62	0.38	0.63	0.30	1.38	1.82	2.01	3.52	0.88	0.40	1.21	1.72	0.0	0.0	0.0	0.0	0.0
K_2O, flame, per cent	1.17	0.99	1.06	0.78	1.98	1.75	1.02	1.72	4.96	2.94	3.82	3.78	1.14	0.61	1.56	1.68	0.0	0.20	0.02	0.0	0.07
Total as Na_2O, flame, per cent	0.97	0.89	0.81	0.77	2.92	1.53	1.30	1.43	4.64	3.75	4.52	6.01	1.63	0.80	2.24	2.83	0.0	0.13	0.01	0.0	0.05
P_2O_5, per cent	0.02	0.02	0.01	0.15	0.23	0.14	0.26	0.51	0.02	0.07	0.11	0.04	0.29	0.14	0.27	0.29	...	...	...	...	...
Mn_2O_3, per cent	0.58	0.53	0.36	0.21	0.07	0.04	0.16	0.04	0.05	0.10	0.05	0.07	0.08	0.05	0.06	0.03	...	...	...	...	...
Insoluble residue, per cent	0.56	0.52	6.5	11.7	70.4	79.4	64.9	76.8	94.6	93.5	61.2	94.9	67.1	86.9	80.3	76.2	...	...	...	...	...
Chloroform solubility, per cent	0.002	0.008	0.002	0.039	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
Moisture content, per cent	0.18	0.13	0.35	0.40	0.20	0.19	0.17	0.26	0.83	0.63	4.3	0.07	1.7	1.2	1.4	4.1	0.07	1.01	0.06	2.90	0.83
Sulfide sulfur, per cent	0.86	1.34	0.06	0.02	0.05	0.00	0.13	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.10	0.09	...	...	...	...	...
Total carbon, per cent	0.45	0.53	...	...	0.43	3.17	11.13	7.22	0.02	0.04	0.08	0.04	1.10	0.07	0.30	0.15	...	...	...	...	...

Fineness:																					
Passing No. 200 sieve, dry, per cent	95.8	98.8	87.9	96.5	95.5	92.8	93.6	85.1	98.6	90.8	86.2	66.9	98.1	70.5	98.8	68.5	...	...	...	...	100.0
Passing No. 325 sieve, wet, per cent	90.2	97.2	91.0	93.2	93.5	87.7	85.8	81.5	98.1	91.4	85.5	67.2	96.8	71.5	98.6	93.0	96.9	96.0	99.9	96.7	98.0
Specific surface, sq cm per g:																					
Blaine air permeability	3 605	3 695	11 260	6 420	3 565	2 940	2 945	4 205	4 410	8 340	10 460	3 415	13 685	11 770	10 450	12 125	4 425	5 500	4 125	476 600	...
Fisher air permeability	3 410	3 390	9 790	5 830	3 335	2 855	2 715	3 960	4 640	8 230	13 215	3 850	12 921	15 020	12 195	15 345	4 545	5 130	4 075	...	...
Klein hydrometer	2 790	4 615	3 835	1 475	7 030	5 960	5 050	5 045	4 505	6 670	9 850	4 085	3 360	7 600	6 485	12 445	2 595	2 790	3 745	...	...
Nitrogen adsorption[b]	9 500	8 100	46 800	26 200	7 900	16 000	24 700	30 200	79 900	60 400	259 300	8 000	242 300	136 000	179 000	259 500	...	...	...	...	...
Particle size (microscope):																					
Minimum, μ	0.5	0.5	−0.5	−0.5	0.5	0.5	1	1	1	0.5	−0.5	−0.5	0.5	−0.5	−0.5	−0.5	0.2	<0.4	0.2	...	...
Maximum, μ	270	400	350	250	1 000	1 000	2 000	3 000	250	500	450	1 000	200	300	400	2 000	100	4	60	...	...
Predominant range, μ	2 to 9	2 to 15	0.5 to 3	0.5 to 3	1 to 10	3 to 13	4 to 20	3 to 14	2 to 20	2 to 14	2 to 8	6 to 15	2 to 6	1 to 7	2 to 10	0.5 to 1	...	...	0.4 to 0.8	...	...
Mean diameter, μ	4.8	4.7	...	...	5.3	7.1	6.9	5.2	4.8	2.5	2.1	6.1	1.6	1.8	2.0	1.8	4.1	2.0	2.2	0.5	...
Specific gravity	2.84	2.87	2.85	2.89	2.48	2.36	2.43	2.26	2.35	2.39	2.27	2.36	2.51	2.35	2.45	2.30	2.64	2.60	2.63	2.20	...
Normal consistency, per cent	30.4	26.2	38.4	36.0	21.8	33.2	47.2	48.0	44.0	43.6	50.8	23.0	25.6	67.0	62.0	36.4	[c]	[c]	[c]	[d]	...
Air content of mortar, per cent	2.8	7.5	4.9	9.6	4.9	2.6	2.7	2.8	2.7	1.7	2.0	4.1	3.2	2.5	3.6	1.6	[c]	5.2	2.5	[d]	...
Bleeding of paste:																					
Rate, ml per sq cm per g	0.000123	0.000117	0.0	0.000018	[e]	0.000119	[f]	[f]	[f]	[f]	[f]	[g]	[f]	[f]	[f]	0.0	[c]	[c]	[c]	[d]	...
Capacity, ml per ml	0.055	0.055	0.0	0.004	[e]	0.023	[f]	[f]	[f]	[f]	[f]	[g]	[f]	[f]	[f]	0.0	[c]	[c]	[c]	[d]	...

---

[a] Values corrected for sulfur.
[b] Tests conducted by National Bureau of Standards.
[c] Insufficient quantity of material to complete all tests.
[d] Material is hygroscopic and gelled.
[e] Accurate bleeding results could not be obtained.
[f] Using the specified proportions of water to solids, resultant pastes were too dry to test.
[g] Bleeding was too great to be measured with available apparatus.

been described **(2, 6, 7)**.[2] In some, only the mineral admixture is involved; in others, the admixture is mixed with either lime or portland cement. Several tests to evaluate the effectiveness of mineral admixtures in preventing excessive expansion due to the alkali-aggregate reaction have been proposed. These tests, as well as those for determination of alkali release and fineness, were selected for use in this investigation. The tests used were evaluated for reliability, and the minimum percentage of the replacement material that would effectively reduce the expansion was determined.

MATERIALS

Portland Cements:

Three portland cements were used in this investigation: two type I cements, one of high alkali content (1.07 per cent alkalies calculated as soda) and one of low alkali content (0.37 per cent); and one type II cement of medium alkali content (0.68 per cent). The results of tests of these cements are given in Table I.

Replacement Materials:

The 20 replacement materials studied represent eight classes. The classes, the specific materials tested, and the abbreviations for the materials used herein, are as follows:

Class of Material	Material
1. Granulated blast-furnace slags	Slag I
	Slag II
2. Natural cements	Natural I (Nat I)
	Natural II (Nat II)
3. Fly ashes	Fly ash I (FA I)
	Fly ash II (FA II)
	Fly ash III (FA III)
	Fly ash IV (FA IV)
4. Natural volcanic glasses	Pumicite (Pum F)
	Pumicite (Pum L)
	Tuff
	Obsidian (Obs)
5. Calcined opaline shales	Calcined shale (C Sh M)
	Calcined shale (C Sh N)
	Calcined diatomaceous earth (Cal D)
6. Uncalcined diatomite	Uncalcined diatomaceous earth (Unc D)
7. Uncalcined quartz	Silica flour
	Air float
	Ad-mix
8. Glass	Synthetic, pure silica glass (SS glass)

The 16 materials in the first six classes were also used in the general investigation of cement-replacement materials. The methods of processing these materials and data on their use in concrete are given in report 1 of that investigation, **(11)** Classes 7 and 8 consist of four materials, all of which may be regarded as essentially pure silica. The three materials in class 7 are quartz and the one in class 8 is an amorphous silica. The results of tests of all 20 materials are given in Table II.

Hydrated Lime:

Some of the tests required the admixtures to be reacted with hydrated lime. The lime selected conformed to ASTM Specifications C 6 for Normal Finishing Hydrated Lime[3] and, in addition, it was required that it contain not less than 75 per cent CaO nor more than 5 per cent MgO based on the nonvolatile portion, and that not more than 5 per cent be retained on the No. 325 sieve. The properties of the lime are given in Table II.

Aggregates:

Three aggregate conditions were used in making 1 by 1 by 11-in. bars for expansion tests: Pyrex glass, Sioux quartzite, and a Sioux-Klufa opaline quartzite combination, all previously described in Waterways Experiment Station reports (10, 12). Both the reactive aggregate (Pyrex glass) and the innocuous aggregate (Sioux quartzite) were graded as required by CRD-C 123 (9). The combination of 90 per cent Sioux quartzite and 10 per cent Klufa opaline quartzite has been found (12) to be more reactive than any other combination of these materials. This combination was graded as follows:

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] 1958 Book of ASTM Standards, Part 4 p. 205.

Sieve	Percentage Retained: Sioux	Klufa
⅜-in. to No. 4	2.5	...
No. 4 to No. 8	7.5	...
No. 8 to No. 16	10.0	...
No. 16 to No. 30	32.0	3.0
No. 30 to No. 50	25.6	4.9
No. 50 to No. 100	9.0	2.0
Passing No. 100	3.5	...
Total	90.1	9.9

TESTS AND RESULTS

Test for Reactivity with Sodium Hydroxide (NaOH):

Test Method.—To evaluate admixtures for their effectiveness in preventing excessive expansion of concrete, an adaptation of the quick chemical test for reactivity of aggregate with sodium hydroxide has been used. Since some materials will harden when 25 g of the admixture is reacted with 25 ml of 1*N* NaOH, it was necessary to reduce the weight of admixture used to 12.5 g. In all other respects the test is the same as described in the Corps of Engineers version of the quick chemical test (CRD-C 128) (9).

Tests of Individual Materials and Blends.—Each of the 19 admixtures in classes 1 through 7 and the two type I portland cements were tested alone. The twentieth admixture, synthetic pure silica glass, was not tested because its great absorptive capacity precluded testing without further modification of the test method. Each material was tested in either duplicate or triplicate.

The average results of the tests of each of the admixtures and the portland cements are given in Table III. The two type I cements gave values for dissolved silica (S_c) of zero and for reduction in alkalinity (R_c) of 187 and 137. The 19 admixtures gave results for S_c ranging from 0 to 883 and for R_c ranging from 65 to 545. The utility of this test would be greatly enhanced if either or both of the determined functions (S_c, R_c) were additive. However, the results obtained from a series of tests definitely indicate that neither function is additive and, therefore, the per-

TABLE III.—RESULTS OF QUICK CHEMICAL TEST.

Material	Cements and Replacement Materials Alone		
	Dissolved, Silica S_c, millimoles per liter	Reduction in Alkalinity, R_c, millimoles per liter	Reduction in Alkalinity R_a per cent
Type I cement, low alkali	0	187	18
Type I cement, high alkali	0	137	14
Slag I	0	74	8
Slag II	0	68	7
Natural cement I	1	119	12
Natural cement II	0	113	11
Fly ash I	44	456	46
Fly ash II	158	280	28
Fly ash III	33	352	36
Fly ash IV	151	246	25
Pumicite F	270	167	17
Pumicite L	359	188	19
Tuff	450	406	41
Obsidian	204	65	7
Calcined shale M	405	510	52
Calcined shale N	883	390	39
Calcined diatomaceous earth	382	545	55
Uncalcined diatomaceous earth	810	430	44
SiO_2 flour	360	248	28
Air float	39	351	38
Ad-mix	82	85	11.6

formance of a given blend cannot be predicted.

Discussion of Results.—Two criteria have been applied to the test to determine the effectiveness of admixtures in reducing expansion due to alkali-aggregate reaction. The Davis Dam specification (8) required a minimum reduction in alkalinity (R_a) of 40 per cent for a material to be regarded as effective. Of the materials tested in this investigation, the data in Table III show that only five meet this requirement: fly ash I, tuff, calcined shale M, calcined diatomite, and uncalcined diatomite, with R_a values of 46, 41, 52, 55, and 44 per cent, respectively. Calcined shale N, with an R_a value of 39 per cent, may be regarded as borderline. The second criterion was proposed by Moran and Gilliland (7) who plotted the S_c and R_c values for a number of materials and found that those that will reduce mortar expansions 75 per cent or more in two weeks at 20 per cent replacement by weight (25 per cent by volume) will fall above or to the right of a line having the equation $R_c + \frac{2}{3} S_c = 630$. The test results in Table III have been graphically represented in Fig. 1. It is evident that calcined diatomite, calcined shale M, tuff, uncalcined diatomite, and calcined shale N meet this requirement. Although fly ash I met the Davis Dam specifications, it does not conform to this requirement. Table IV summarizes these data as well as the data obtained by other methods used in this investigation to test the effectiveness of mineral admixtures in preventing expansion due to alkali-aggregate reaction.

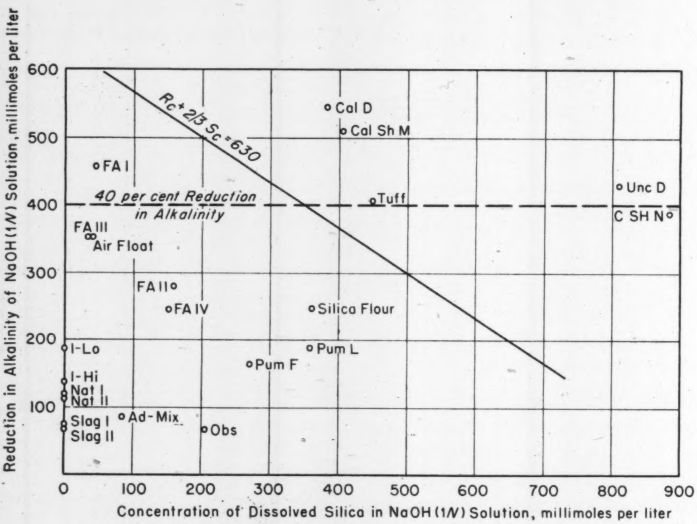


FIG. 1.—Reactivity of Materials with Sodium Hydroxide.

Modified Test for Reactivity with NaOH:

Modified Method.—The Bureau of Reclamation used the quick chemical test for some time to assist in the evaluation of admixtures (8). However, it did

TABLE IV.—SUMMARY OF TEST RESULTS ON EFFECTIVENESS OF ADMIXTURES.

Note.—The letter *n* denotes that the admixture was found to be noneffective, the letter *e* that the admixture was found to be effective.

Admixture	Volume Replacement Material, per cent	Reactivity with $Na^+ (OH^-)$[a]		Modified Reactivity with NaOH			Mortar-Bar Reduction in Expansion > 75 per cent		
				Na^+ plus K^+ moles, per liter > 180	OH^- moles per liter				
		$R_a \geq$ 40 per cent	$R + 2/3\ S \geq 630$		Single End Point > 210	Double End Point > 210	14 days	6 months	1 year
Slag I	70	...	...	*n*	*n*	*n*	...	...	...
	50	...	...	*n*	*n*	*n*	*e*	*e*	*e*
	30	...	...	*n*	*n*	*n*	...	...	...
	25	*n*	*n*	...	...	...	*n*	*n*	*n*
Slag II	50	...	...	*n*	*n*	*n*	*e*	*e*	*e*
	25	*n*	*n*	...	...	...	*n*	*n*	*n*
Fly ash I	60	...	...	*n*	*e*	*e*	...	...	...
	45	...	...	*n*	*e*	*e*	*n*	*e*	*e*
	30	...	...	*n*	*n*	*e*	...	...	...
	22.5	*e*	*n*	...	...	...	*n*	*n*	*n*
Fly ash II	45	...	...	*n*	*n*	*n*	*n*	*e*	*e*
	22.5	*n*	*n*	...	...	...	*n*	*n*	*n*
Fly ash III	45	...	...	*n*	*n*	*n*	*n*	*e*	*e*
	22.5	*n*	*n*	...	...	...	*n*	*n*	*n*
Fly ash IV	45	...	...	*n*	*n*	*n*	*e*	*e*	*e*
	22.5	*n*	*n*	...	...	...	*n*	*n*	*n*
Pumicite F	45	...	...	*e*	*e*	*e*	...	...	...
	35	...	...	*n*	*n*	*n*	*n*	*e*	*e*
	25	*n*	*n*	*n*	*n*	*n*	...	...	...
	17.5	...	...	...	...	...	*n*	*n*	*n*
Pumicite L	35	...	...	*n*	*e*	*e*	*e*	*e*	*e*
	17.5	*n*	*n*	...	...	...	*n*	*n*	*n*
Tuff	35	...	...	*e*	*e*	*e*	*n*	*e*	*n*
	17.5	*e*	*e*	...	...	...	*n*	*n*	*n*
Obsidian	35	...	...	*n*	*n*	*n*	*n*	*e*	*e*
	17.5	*n*	*n*	...	...	...	*n*	*n*	*n*
Calcined shale M	40	...	...	*e*	*e*	*e*	...	...	...
	30	*e*	*e*	*e*	*e*	*e*	*e*	*e*	*e*
	20	...	...	*n*	*n*	*n*	...	...	...
	15	...	...	...	...	...	*n*	*n*	*n*
Calcined shale N	30	*n*	*e*	*e*	*e*	*e*	*e*	*e*	*e*
	15	...	...	...	...	...	*n*	*n*	*n*
Calcined diatomaceous earth	30	*e*	*e*	*e*	*e*	*e*	*n*	*e*	*e*
	15	...	...	...	...	...	*n*	*n*	*n*
Uncalcined diatomaceous earth	16	*e*	*e*	*n*	*n*	*n*	...	...	...
	12	...	...	*n*	*n*	*n*	*n*	*n*	*n*
	8	...	...	*n*	*n*	*n*	...	...	...
	6	...	...	...	...	...	*n*	*n*	*n*

---

[a] In this test, the admixture is used by itself. The criterion correlates the results with mortar-bar results in which 20 per cent portland cement by weight (25 per cent by volume) is replaced by the admixture.

not prove to be completely satisfactory, and in 1952, a description of a modified version of the reactivity test (6) was published. The modified test assumes an admixture-to-cement ratio of 1 to 3 by weight and a tricalcium silicate (C_3S) content of cement of 40 per cent. Therefore, 25 ml of 0.5N NaOH (instead of 25 ml of 1N NaOH) are reacted with 4.0 g of admixture and 1.5 g of calcium hydroxide ($Ca(OH)_2$). The reaction container, the reaction time, and the temperature are the same as for the original method. The containers are, however, agitated throughout the reaction period by an end-over-end rotation at 30 rpm. In addition to the determination of S_c and R_{OH}[4] on the filtrate, the net reduction in concentration of sodium (Na) and potassium (K) was determined and also the concentration of alumina (Al_2O_3).

Criteria.—The paper by Mielenz *et al* (6) correlated graphically the reduction in alkalies (R_A) and the reduction in alkalinity (R_{OH}) of 63 admixtures representing a wide variety of materials, with the reduction in expansion (R_e) of mortar bars containing Pyrex aggregate, high-alkali cement, and the tested admixture at ages of 14 days, 2 months, and 1 year. The correlations were found to be satisfactory for all three ages, but best at 14 days; therefore, only the 14-day correlations were shown. Admixtures that reduced expansion by 75 per cent in the mortar-bar test were generally regarded as effective. The value of 75 per cent reduction was, therefore, used to determine the criteria for the chemical tests. The values of 180 or greater for reduction in alkalies and 210 or greater for reduction in alkalinity were apparently selected from the graph as being those that will most efficiently separate admixtures that will reduce mortar-bar expansion by 75 per cent or more from those that will reduce expansion by less than 75 per cent.

Statistical Determination of Criteria.—The published data did not include the line of best fit, which may substantiate the criteria. As part of this investigation, the equations of these lines, the correlation coefficients, and the standard errors of estimate (4) were calculated and are:

Determination	Test Age	Equation of Regression Line.[a] R_e	Correlation Coefficient	Standard Error of Estimate	Value Corresponding to 75 per cent Reduction in Expansion
Net reduction of alkalies	14 days	$0.162\ R_A + 41.0$	0.84	12.5	209.7
	2 months	$0.161\ R_A + 41.0$	0.79	15.3	211.3
	1 year	$0.165\ R_A + 33.2$	0.78	16.6	253.1
Reduction in alkalinity	14 days	$0.154\ R_{OH} + 38.2$	0.84	12.4	238.6
	2 months	$0.156\ R_{OH} + 37.6$	0.82	14.3	239.7
	1 year	$0.165\ R_{OH} + 29.4$	0.82	15.3	276.5

[a] R_A = reduction in alkalies, milliequivalents per liter,
R_{OH} = reduction in alkalinity, milliequivalents per liter, and
R_e = per cent reduction in mortar expansion.

The differences between the equations for the three test ages and between the corresponding correlation coefficients are not significant. The magnitude of the correlation coefficients indicates that the regression lines are not due to random chance. Information concerning the reproducibility of the test was included with the published data. The standard deviation for reduction in alkalies was found to vary between 7.4 and 14.5. The criterion specified for reduction in alkalies is two to three standard deviations less than the values found by use of the regression line to correspond to a 75 per cent reduction in expansion both at 14 days and 2 months. The criterion for reduction in alkalies, and probably for reduction in alkalinity, is too low in that many admixtures that will reduce expansion by only 70 per cent in 14 days will be evaluated as effective along with most if not all admixtures that will reduce expansion by 75 per cent or more. The criteria apparently take into account the lack of precision of the chemical test without considering the lack of precision of the mortar-bar test.

---

[4] In this paper, the reduction in alkalinity as determined in the modified test is designated R_{OH} rather than R_c.

TABLE V.—RESULTS OF MODIFIED QUICK CHEMICAL TEST.

Material	Replacement, per cent	Blends Made with $Ca(OH)_2$ Equivalent of Type I High-Alkali Cement, milliequivalents per liter					Blends Made with $Ca(OH)_2$ Equivalent of Type I Low-Alkali Cement, milliequivalents per liter				
		Reduction in Na^+	K^+ Released	Net Reduction in Alkali, $(Na^+ + K^+)$	Reduction in Alkalinity (OH^-)		Reduction in Na^+	K^+ Released	Net Reduction in Alkali, $(Na^+ + K^+)$	Reduction in Alkalinity (OH^-)	
					Single End Point	Double End Point				Single End Point	Double End Point
Cement	0	6	0	6	−2	−4	5	0	5	5	1
Slag I	30	34	10	24	16	32	−4	19	−23	49	73
	50	25	6	19	35	59					
	70	10	13	−3	90	142					
Slag II	50	162	8	154	37	54	...	...	...	...	...
Natural cement I	20	8	19	−11	−5	−19	−16	28	−44	41	32
	35	3	24	−21	45	32					
	50	−19	−39	−58	70	73					
Natural cement II	35	−12	21	−32	35	31	...	...	...	...	...
Fly ash I	30	69	13	56	167	223	60	19	41	239	312
	45	52	22	30	261	327					
	60	87	13	75	295	376					
Fly ash II	45	134	18	116	170	200	...	...	...	...	...
Fly ash III	45	52	11	41	148	182	...	...	...	...	...
Fly ash IV	45	110	15	95	146	198	...	...	...	...	...
Pumicite F	25	60	45	15	104	121	140	49	91	116	144
	35	152	48	104	154	185					
	45	220	26	194	227	256					
Pumicite L	35	199	24	175	235	265	...	...	...	...	...
Tuff	35	224	16	208	257	293	...	...	...	...	...
Obsidian	35	47	33	14	50	73	...	...	...	...	...
Calcined shale M	20	50	2	48	68	72	151	6	145	175	172
	30	216	0	216	238	243					
	40	325	−6	331	377	381					
Calcined shale N	30	356	0	356	386	390	...	...	...	...	...
Calcined diatomaceous earth	30	258	6	252	355	362	...	...	...	...	...
Uncalcined diatomaceous earth	8	47	0	47	−2	−6	−17	8	−25	36	23
	12	43	4	39	44	37					
	16	95	−2	97	53	72					

Additional Modification of the Test.—An additional series of tests was made in which the mixtures of replacement material and calcium hydroxide that were reacted with NaOH were proportioned to represent specific admixture-to-cement ratios by solid volume and specific calculated C_3S contents rather than the assumed value of 40 per cent. In this series, the SiO_2 and Al_2O_3 determinations were omitted and the double end point titration was used.

Blends Tested.—The reactivity of admixture-calcium hydroxide mixtures representing the following blends was determined by the procedure just described.

Class	Replacement Material	Replacement, Solid Volume,[a] per cent	
		Type I High-Alkali	Type I Low-Alkali
1......	Slag I	30, 50, 70	50
	Slag II	50	..
2......	Nat I	20, 35, 50	35
	Nat II	35	..
3......	FA I	30, 45, 60	45
	FA II	45	..
	FA III	45	..
	FA IV	45	..
4......	Pum F	25, 35, 45	35
	Pum L	35	..
	Tuff	35	..
	Obs	35	..
5......	C Sh M	20, 30, 40	30
	C Sh N	30	..
	Cal D	30	..
6.......	Unc D	8, 12, 16	12

[a] Values taken from reference (11).

Test Results.—Test results obtained for these blends and for the lime substituting for the two cements are shown in Table V. All the results shown were obtained from duplicate determinations.

The following blends, of all those tested, were found to meet both published criteria (6) when tested with the calcium hydroxide ratio representing the high-alkali cement: 45 per cent pumicite F, 35 per cent tuff, 30 and 40 per cent calcined shale M, 30 per cent calcined shale N, and calcined diatomite. None of the blends made with uncalcined diatomite were found to meet the requirements; undoubtedly, the percentage of uncalcined diatomite used was too low. Some anomalies were also observed. The 35 per cent blend of pumicite L with high-alkali cement gave results greater than 210 for reduction in alkalinity but the results for reduction in alkalies were less than 180; this was also found for the 45 and 60 per cent of fly ash I with high-alkali cement and the 45 per cent blend with low-alkali cement. The 30 per cent blend of fly ash I with high-alkali cement met the 210 criteria only when the double end point titration was used. Only the 45 per cent blend of pumicite F with high-alkali cement would be affected if the criteria were changed to those derived from the regression line. The other blends (35 per cent tuff, 30 and 40 per cent calcined shale M, 30 per cent calcined shale N, 30 per cent calcined diatomite, all with high-alkali cement) would still be regarded as effective inhibitors. The following general observations were apparent from this test:

(*a*) The amount of replacement material used must exceed a given minimum before expansion can be effectively reduced and, for the materials tested, the minimum replacement was never lower than 30 per cent.

(*b*) The values for reduction in alkalies (R_A) and alkalinity (R_{OH}) generally increased with increasing replacement.

(*c*) Blends made with low-alkali cement will not necessarily give results similar to those obtained with blends made with high-alkali cement.

(*d*) Changing the numerical values of the criteria does not materially affect the interpretation of the results.

Mortar-Bar Test:

Mortars.—The only direct method available for determining the degree of expansion caused by alkali-aggregate reaction is the mortar-bar test. Similarly, the only direct method available for determining the effectiveness of an admixture in preventing excessive expansion due to the reaction is to compare the expansion of bars made with the admixture and a reactive aggregate with bars made without the admixture (control bars), all other conditions being the same. For this phase of the investigation, each of the 14 materials in classes 1 and 3 through 6 and the one admixture in class 8 were used both at the assumed optimum and at one half the assumed optimum percentages by solid volume (the class 8 material was, in addition, used at one fourth optimum) with the type I, high-alkali portland cement. Three sets of three bars each, each set made on a different date, were made for each blend with Pyrex glass as the aggregate. The bars were fabricated in accordance with CRD-C 123 (9) except that:

(*a*) The water content of the mortar was adjusted for a flow of 110 ± 5 per cent by ten $\frac{1}{2}$-in. drops in 6 sec.

(*b*) The quantity of cementing material used per batch did not equal 450 g; however, the volume of cementing material used per batch did equal that of 450 g of portland cement.

Method CRD-C 123 (9), with the exceptions noted above, differs from ASTM Method C 227[5] from which mortar-bar methods used by other laboratories were derived in the following essential respects:

(*a*) The grading of the Pyrex aggregates differs as follows:

Sieve Size		Percentage Used	
Passing	Retained	CRD-C 123	ASTM C 227
$\frac{3}{8}$-in.	No. 4	2.5	0
No. 4	No. 8	7.5	20
No. 8	No. 16	10.0	20
No. 16	No. 30	35.0	20
No. 30	No. 50	30.5	20
No. 50	No. 100	11.0	20
No. 100	Pan	3.5	0

(*b*) CRD-C 123 provides for stripping bars from molds at an age of 44 ± 4 hr while ASTM Method C 227 provides for stripping at 24 ± 2 hr.

In addition, three sets of bars were made with the first admixture in each of the above classes at the assumed optimum (11) percentage only (class 8 material was also blended at one half optimum percentage), blended with the type I high-alkali cement and with the Sioux-Klufa combination as the aggregate. Finally, these same admixtures at the same percentage replacement were blended with the type II medium-alkali cement, and bars were made with Pyrex glass as the aggregate. The following control mixtures were used, each represented by three sets of three bars.

(*a*) Type I high-alkali cement, Pyrex glass.

(*b*) Type I high-alkali cement, Sioux-Klufa combination.

(*c*) Type I high-alkali cement, Sioux quartzite.

(*d*) Type II medium-alkali cement, Pyrex glass.

(*e*) Type II medium-alkali cement, Sioux-Klufa combination.

Tests and Results.—The bars were stored for a period of 1 yr. Their length change was determined at 14 days, 6 months, and 1 yr according to Method CRD-C 123. The percentage reduction

---

[5] Tentative Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227–58T), 1958 Book of ASTM Standards, Part 4, p. 514.

TABLE VI.—RESULTS OF MORTAR-BAR EXPANSION TESTS.

Replacement Material		Expansion, per cent			Reduction in Expansion, per cent			Warp,[a] per cent	Petrographic Rating of Bar Condition
Type	Volume, per cent	14 days	6 months	1 yr	14 days	6 months	1 yr	1 yr	
TYPE I HIGH-ALKALI CEMENT									
Pyrex Aggregate									
None	...	0.106	0.221	0.230	...	...	...	0.05	2
Slag I	50	0.008	0.011	0.014	92	95	94	0.0	1
	25	0.060	0.098	0.102	43	56	56	0.03	2
Slag II	50	0.021	0.025	0.028	80	89	88	0.02	2
	25	0.084	0.117	0.123	21	47	47	0.04	2
Fly ash I	45	0.028	0.033	0.035	74	85	85	0.0	1
	22.5	0.063	0.089	0.093	41	60	60	0.0	2
Fly ash II	45	0.031	0.032	0.037	71	86	84	0.0	1
	22.5	0.069	0.090	0.095	35	59	59	0.0	1
Fly ash III	45	0.034	0.035	0.037	68	84	84	0.0	1
	22.5	0.059	0.086	0.093	44	61	60	0.0	1
Fly ash IV	45	0.027	0.026	0.028	75	88	88	0.0	1
	22.5	0.063	0.083	0.091	41	62	60	0.0	1
Pumicite F	35	0.035	0.040	0.045	67	82	80	0.0	2
	17.5	0.059	0.082	0.087	44	63	62	0.0	2
Pumicite L	35	0.025	0.034	0.040	76	85	83	0.0	1
	17.5	0.065	0.088	0.094	39	60	59	0.02	1
Tuff	35	0.028	0.052	0.060	74	76	74	0.0	1
	17.5	0.072	0.155	0.170	32	30	26	0.01	2
Obsidian	35	0.036	0.046	0.053	66	79	77	0.0	1
	17.5	0.081	0.127	0.136	24	43	41	0.03	2
Calcined shale M	30	0.018	0.021	0.029	83	90	87	0.0	1
	15	0.069	0.092	0.104	35	58	55	0.0	1
Calcined shale N	30	0.004	0.002	0.009	96	99	96	0.0	1
	15	0.044	0.057	0.067	58	74	71	0.02	1
Calcined diatomaceous earth	30	0.032	0.037	0.043	70	83	81	0.0	1
	15	0.071	0.104	0.110	33	53	52	0.0	1
Uncalcined diatomaceous earth	12	0.077	0.123	0.134	27	44	42	0.03	1
	6	0.101	0.185	0.198	5	16	14	0.03	1
Synthetic silica glass	10	0.024	0.053	0.086	77	76	63	0.03	1
	5	0.059	0.182	0.236	44	18	−3	0.04	1
	2.5	0.101	0.260	0.293	5	−18	−27	0.05	2

TABLE VI.—*Concluded.*

Replacement Material		Expansion, per cent			Reduction in Expansion, per cent			Warp,[a] per cent	Petrographic Rating of Bar Condition
Type	Volume, per cent	14 days	6 months	1 yr	14 days	6 months	1 yr	1 yr	
Sioux-Klufa Aggregate									
None	...	0.159	0.681	0.803	...	...	...	0.06	5
Slag I	50	0.010	0.117	0.180	94	83	78	0.0	3
Fly ash I	22.5	0.065	0.381	0.464	59	44	42	0.04	3
Pumicite F	35	0.018	0.282	0.361	89	59	55	0.04	3
Calcined shale M	30	0.018	0.051	0.102	89	93	87	0.02	3
Uncalcined diatomaceous earth	12	0.119	0.792	0.964	25	−16	−20	0.05	5
Synthetic silica glass	10	0.021	0.044	0.059	87	94	93	0.01	2
	5	0.053	0.281	0.317	67	59	61	0.03	3
Sioux Aggregate									
None	...	0.004	0.027	0.057	...	...	...	0.0	2
TYPE II MEDIUM-ALKALI CEMENT									
Pyrex Aggregate									
None	...	0.046	0.068	0.075	...	...	...	0.0	2
Slag I	50	−0.003	−0.009	−0.005	107	113	107	0.0	1
Fly ash I	45	0.008	0.005	0.008	83	93	89	0.0	1
Pumicite F	35	−0.002	−0.004	0.003	104	106	96	0.0	1
Calcined shale M	30	0.000	−0.005	−0.002	100	107	103	0.0	1
Uncalcined diatomaceous earth	12	0.001	0.002	0.004	98	97	95	0.0	1
Synthetic silica glass	10	0.008	−0.019	−0.022	83	128	129	0.0	1
	5	0.006	0.004	0.016	87	94	79	0.0	1
Sioux-Klufa Aggregate									
None	...	0.100	0.454	0.537	...	...	...	0.02	4

[a] Determined according to Method CRD-C 123.

in expansion was calculated by the following equation:

$$R_e = \frac{E_r - E_t}{E_r} \times 100$$

where:

R_e = reduction in mortar expansion, per cent,

E_t = average expansion of bars containing the admixture, per cent, and

E_r = average expansion of the applicable control bars, per cent.

The average results of all these tests are shown in Table VI both as percentage expansion and percentage reduction in expansion at bar ages of 14 days, 6 months, and 1 year.

Discussion of Test Results.—The expansion due to alkali-aggregate reaction was reduced for every combination tested except in three cases:

the bars made from mortars containing blends of high-alkali cement, both 2.5 and 5.0 per cent of the synthetic silica glass with Pyrex glass aggregate (¼ and ½ optimum percentages of the admixtures), and the blend of 12 per cent uncalcined diatomite with the Sioux-Klufa aggregate. The degree of reduction of the expansion was increased by increasing the percentages of admixture in the mortar. Maximum reduction of expansion for bars made with Pyrex glass aggregate was found for the blend of 30 per cent calcined shale N with high-alkali cement. For mortars made with the Sioux-Klufa combination as the aggregate, the maximum reduction in expansion was found for the blend containing the synthetic silica glass at its optimum percentage. Three of the mortars made with the medium-alkali cement showed shrinkage at the age of 1 yr: those containing calcined shale M, slag I, and synthetic silica glass at their optimum percentages.

The following materials may be regarded as effective at a test age of 14 days, using the criterion of a reduction in expansion of at least 75 per cent when tested in mortars made with high-alkali cement and Pyrex glass: slags I and II, fly ash IV, pumicite L, calcined shale M, calcined shale N, and synthetic silica glass at their optimum percentage replacements. At test ages of 6 months and 1 yr, all the admixtures tested (at their optimum percentage replacement), except uncalcined diatomite, caused reduction in expansion of 75 per cent or more. When the Sioux-Klufa combination was used as the aggregate, slag I, calcined shale M, and synthetic silica caused reduction in expansion of 75 per cent or more at all three ages and pumicite F at a test age of 14 days. All the admixtures tested with the medium-alkali cement (Pyrex glass-aggregate) caused a reduction in expansion of 75 per cent or greater at all three ages.

Petrographic Examination of Bars.—After expansion measurements were completed, condition of the bars was determined by examination of the exterior and interior surfaces, both visually and with a stereoscopic microscope. Each bar was rated numerically on a condition scale depending on the presence and amount of: (*a*) external signs of reaction such as warping, surface gel, and surface cracking and (*b*) internal signs of reaction, such as gel and reacted aggregate. The scale is as follows:

0 = No signs of reaction, bar as cast
1 = Slight evidence of reaction
2 = Slight evidence of reaction, signs are more abundant
3 = Moderate reaction, some damage
4 = Intermediate between conditions 3 and 5
5 = Severe reaction, damage, and deterioration

The numerical ratings of the bars are given in Table VI. The observations are summarized below:

(*a*) The general observations included the following:

1. There was a definite over-all relationship between the observed condition and the expansion of the bars examined. The actual relationship was that the observable signs of alkali-aggregate reaction increased as the expansion increased.

2. Every bar examined showed some evidence of alkali-aggregate reaction.

3. Calcium sulfoaluminate was not found in any of the bars.

4. All bars containing slag had the typical blue-green color in the interior and across the bottom of the bar as cast. The area covered by the color varied directly with the slag content. The color faded on exposure to air, but was still quite apparent after two months exposure. In a previous investigation of portland blast-furnace slag cements (13), the blue-green color

was found to fade more rapidly than it did in these bars.

TABLE VII.—RATE OF ALKALI RELEASE.

Replacement Material	Alkali Content,[a] per cent of Replacement Material			Water-soluble Alkali Content, per cent of Replacement Material			Total Alkali as Na_2O Released									
							Per Cent of Replacement Material					Per Cent of Alkali Content				
	Na_2O	K_2O	Total[b]	Na_2O	K_2O	Total[c]	Initial	2 days	7 days	28 days	90 days	Initial	2 days	7 days	28 days	90 days
Slag I	0.20	1.17	0.97	0.00	0.01	0.01	0.00	0.21	0.30	0.37	0.35	0	22	31	38	36
Slag II	0.24	0.99	0.89	0.00	0.00	0.00	0.00	0.17	0.21	0.22	0.19	0	19	24	25	21
Natural cement I	0.11	1.06	0.81	0.01	0.31	0.22	0.23	0.28	0.36	0.49	0.54	28	35	44	60	67
Natural cement II	0.26	0.78	0.77	0.02	0.11	0.09	0.11	0.17	0.27	0.41	0.45	14	22	35	53	58
Fly ash I	1.62	1.98	2.92	0.23	0.04	0.26	0.24	0.28	0.52	1.31	1.20	8	10	18	45	41
Fly ash II	0.38	1.75	1.53	0.03	0.02	0.04	0.04	0.08	0.18	0.56	0.68	3	5	12	37	48
Fly ash III	0.63	1.02	1.30	0.02	0.01	0.03	0.03	0.07	0.16	0.48	0.62	2	5	12	37	48
Fly ash IV	0.30	1.72	1.43	0.01	0.01	0.02	0.02	0.07	0.14	0.35	0.50	1	5	10	24	35
Pumicite F	1.38	4.96	4.64	0.01	0.02	0.02	0.01	0.14	0.46	0.74	0.91	0	3	10	16	20
Pumicite L	1.82	2.94	3.75	0.02	0.01	0.03	0.05	0.18	0.52	0.71	0.56	1	5	14	19	15
Tuff	2.01	3.82	4.52	0.02	0.03	0.04	0.75	1.09	1.08	1.13	1.11	17	24	24	25	24
Obsidian	3.52	3.78	6.01	0.01	0.00	0.01	0.01	0.19	0.51	0.97	1.11	0	3	8	16	18
Calcined shale M	0.88	1.14	1.63	0.02	0.02	0.03	0.04	0.07	0.09	0.13	0.07	2	4	6	8	4
Calcined shale N	0.40	0.61	0.80	0.00	0.02	0.01	0.00	0.03	0.12	0.08	0.05	0	4	15	10	6
Calcined diatomaceous earth	1.21	1.56	2.24	0.03	0.01	0.04	0.03	0.18	0.34	0.37	0.33	1	8	15	16	15
Uncalcined diatomaceous earth	1.72	1.68	2.83	0.04	0.00	0.04	0.02	0.02	0.12	0.10	0.10	1	1	4	4	4
SiO_2 Flour	0.00	0.00	0.00	...	...	...	...	...	...	0.00	...	...	...	...	...	...
Air Float	0.00	0.20	0.13	...	...	...	...	...	...	0.00	...	...	...	...	...	...
Ad-Mix	0.00	0.02	0.01	...	...	...	...	...	...	0.00	...	...	...	...	...	...
Synthetic silica glass	0.00	0.00	0.00	...	...	...	...	...	...	0.00	...	...	...	...	...	...

[a] Determined as described in Method CRD-C 244.

[b] Total alkali as $Na_2O = Na_2O + 0.658\ K_2O$.

[c] Determined as described in Method CRD-C 209 (ASTM Methods C 114 – 53, 1958 Book of ASTM Standards, Part 4, p. 63).

(*b*) Specific relationships observed were:

1. All the bars made with Pyrex glass as the aggregate were rated as being of condition 1 or 2. Of the 14 admixtures in classes 1 and 3 through 6, ten had the same rating at both the optimum and half optimum percentages replacement. The other four were rated as showing more reaction with increasing expansion.

2. The bars made with the Sioux-Klufa combination as the aggregate show, in general, higher expansion and higher ratings, varying from 2 to 5, than those made with Pyrex glass aggregate.

3. Both the condition scale rating and the expansion indicate that uncalcined diatomite is not effective when used as a 12 per cent replacement of high-alkali cement with the Sioux-Klufa combination aggregate.

4. All the bars made with the synthetic silica glass required such large amounts

of water in mixing that they were abnormally weak. This made it impossible to use bar strength which normally diminishes with increased reaction, in arriving at rating conditions.

Rate of Alkali Release:

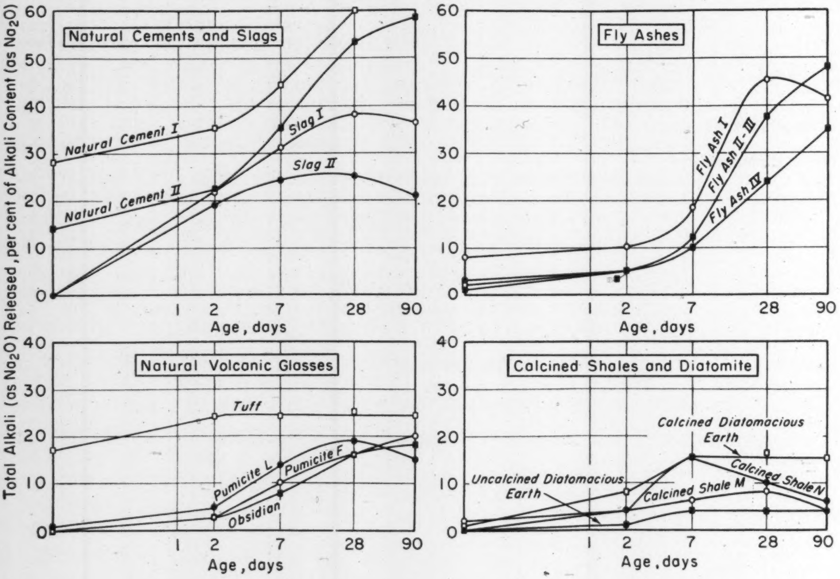


FIG 2.—Rate of Alkali Release from Replacement Materials.

In addition to the other chemical tests described previously, the rate of alkali release of the replacement materials in the presence of lime and water was determined by the method described in Appendix B of the paper by Moran and Gilliland (7). The concentration of the alkalies in solution was determined at 0, 2, 7, 28, and 90 days for each of the 16 materials in classes 1 to 6 and for the 4 materials in classes 7 and 8 at 28 days only. The alkali content of each material was also determined in accordance with CRD-C 244 (9) as well as the water-soluble alkalies as directed in CRD-C 209 (9) The results of all these analyses are shown in Table VII. The alkali content of the solution at various test ages is reported in terms of total alkali, as Na_2O, as percentage of the replacement material, and also as a percentage of the alkali content of the material. The rate of release curves are similar for both sets of data and are shown in Fig. 2, based on the alkali content. The rate of alkali release of the majority of the admixtures increased after the second day, becoming zero or decreasing after 28 days. The diatomites ceased releasing alkali after 7 days, and calcined shale N absorbed alkali at a uniform rate after 7 days. For slags I, II, and calcined shale M, the rate of release was constant up to 28 days and then became negative. Tuff stopped releasing alkali after the second day. The amount of alkali released, as a percentage of alkali content, was greatest for the natural cements, then the fly ashes, followed by the slags and volcanic glasses. The calcined earths released the least alkali. As a percentage of the replacement material, the greatest amount of alkali was released by the volcanic glasses and fly ash I. The least amount was released by the calcined earths and the slags.

Mean Particle Diameter:

In addition to the determination of particle size by microscopic methods (Table II), the mean particle diameter for each of the replacement materials was calculated as described in ASTM Specification C 402 - 58 T, Section 8(*b*).[6] The calculated values are shown in Tables II and VIII and correspond very well to the microscopic results.

TABLE VIII.—COMPARISON OF EFFECTIVENESS OF ADMIXTURES AND CORRELATION OF MINIMUM REPLACEMENT VALUES WITH PHYSICAL PROPERTIES OF ADMIXTURE.

Replacement Material	Minimum per cent Replacement, Solid Volume, for Effective Inhibition	Mean Diameter Calculated, μ	Predominant Range, Microscope, μ	Total Alkali as Na_2O, Released, per cent of Alkali Content		S_c Dissolved Silica, millimoles per liter
				28 days	90 days	
Synthetic silica glass	10	0.5				
Calcined shale N	19	1.8	1 to 7	10	6	883
Uncalcined diatomaceous earth	22	1.8	0.5 to 1	4	4	810
Calcined shale M	26	1.6	2 to 6	8	4	405
Calcined diatomaceous earth	29	2.0	2 to 10	16	15	382
Pumicite L	32	2.5	2 to 14	19	15	359
Pumicite F	35	4.8	2 to 20	16	20	270
Tuff	36	2.1	2 to 8	25	24	450
Obsidian	36	6.1	6 to 15	16	18	204
Slag I	39	4.8	2 to 9	38	36	0
Fly ash IV	40	5.2	3 to 14	24	35	151
Fly ash I	41	5.3	1 to 10	45	41	44
Fly ash II	42	7.1	3 to 13	37	48	158
Fly ash III	44	6.9	4 to 20	37	48	33
Slag II	45	4.7	2 to 15	25	21	0

Autoclave Expansion:

Pastes containing the type II cement and the replacement materials in classes 1, and 2 through 8, in the proportion of 4 to 1 were tested for autoclave expansion. All ten bars tested were sound; the maximum expansion was 0.13 per cent for pumicite L.

COMPARISON OF TEST RESULTS AND CORRELATIONS

Comparison of Chemical and Mortar-Bar Test Results:

Most of the results of the various tests previously described are compared in Table IV. Only the results concerning materials in classes 1 and 3 through 6 are shown. For those tests involving blends of an admixture and a cement or an admixture in a mortar, only the high-alkali cement and the Pyrex glass aggregate results are shown. In addition, the criteria were rigidly interpreted to determine whether a material would be effective; that is, when a test result was found to be one unit less than the criterion, the admixture was judged to be noneffective. As can be seen from the table, complete agreement between the methods was

---

[6] Tentative Specifications for Raw or Calcined Natural Pozzolans for Use as Admixtures in Portland Cement Concrete (C 402-58T), 1958 Book of ASTM Standards, Part 4, p. 519.

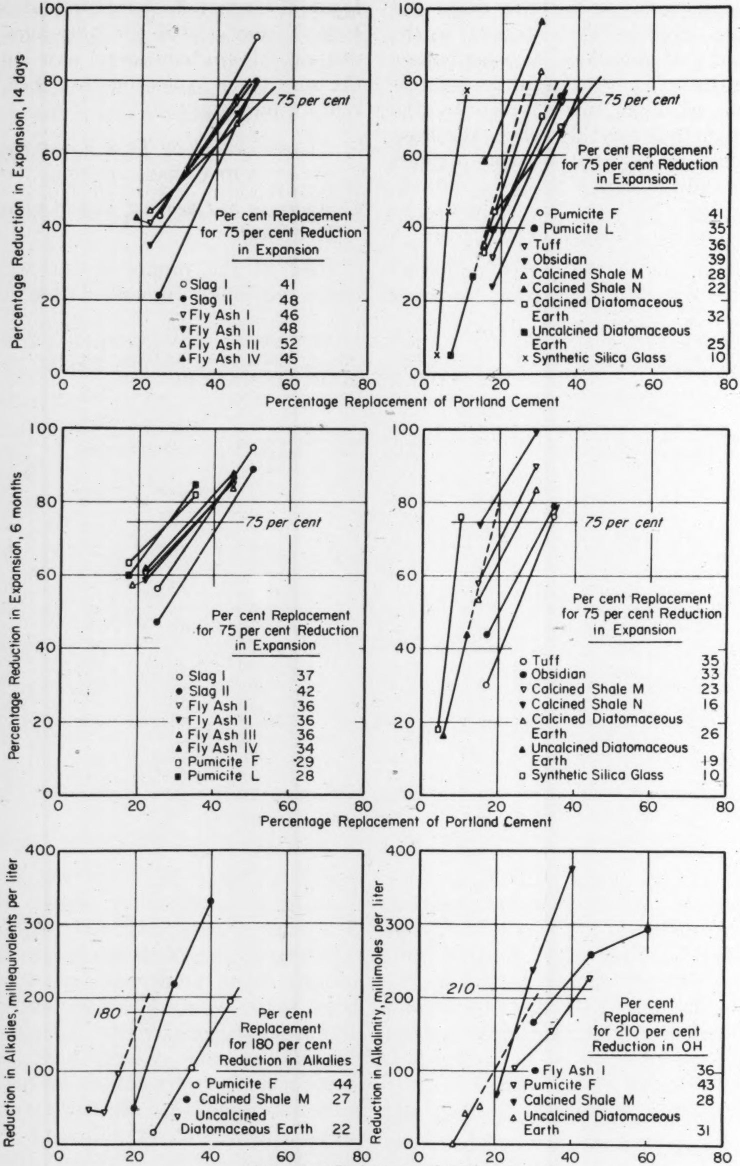


FIG. 3.—Determination of Minimum Percentage Replacement of Cement for Reduction in Expansion, Alkalies, and Alkalinity.

obtained only for calcined shale M, which is effective as a replacement of 25 per cent by volume or greater. If it is assumed that the percentage replacement used for uncalcined diatomite was too low, the methods agree and uncalcined diatomite will not be effective as replacements of 16 per cent or less. It was noted previously and is also evident in Table IV that all the materials tested, except uncalcined diatomite, caused reduction in expansion of 75 per cent or greater at test ages of 6 months and 1 yr when used at their "optimum" percentage replacement. If the 6-month and 1-yr test results are not considered, agreement between the methods was also obtained for fly ash II, fly ash III, obsidian (all noneffective at their optimum percentage replacement or lower), pumicite F (effective at 45 per cent replacement but not at the optimum or lower), and calcined shale N (found effective at optimum percentage replacement but disagreement was found between the two criteria for reactivity with NaOH at 25 per cent replacement). Slags I and II and fly ash IV were found effective at their optimum percentage replacement by the mortar-bar test but ineffective by any of the chemical tests. Tuff and calcined diatomite were found to be effective by the chemical tests at their optimum percentage replacement but ineffective by the mortar-bar tests at 14-day test age. The lack of agreement in the results for fly ash I and pumicite L is between the chemical methods.

Minimum Percentages of Replacement for Effectiveness:

For the proper employment of a criterion of effectiveness based on the ability of an admixture to reduce expansion by a given proportion, or to a given proportion, of that which takes place when no admixture is used, it is necessary that the control condition be one in which substantial expansion takes place. It further follows that the effectiveness of the admixture must be evaluated under conditions in which it serves a function other than that of a mere diluent. It has been shown for a given case studied by U. W. Stoll at the U. S. Naval Civil Engineering Laboratories that, for a reactive aggregate used with a cement of 1.2 per cent alkalies calculated as soda, substitution of a cement having 0.50 per cent alkalies would result in 75 per cent less expansion and that replacement of 60 per cent of the cement of 1.2 per cent alkali content with an inert diluent should be expected to produce the same reduction in expansion.

TABLE IX.—MINIMUM PERCENTAGE REPLACEMENT BY VOLUME FOR EFFECTIVENESS.

Replacement Material	Mortar-Bar Test			Modified Reactivity with NaOH	
	14 days	6 months	Average	Reduction in Alkalies	Reduction in Alkalinity
Slag I	41	37	39	...	...
Slag II	48	42	45	...	...
Fly ash I	46	36	41	...	36
Fly ash II	48	36	42	...	...
Fly ash III	52	36	44	...	...
Fly ash IV	45	34	40	...	...
Pumicite F	41	29	35	44	43
Pumicite L	35	28	32	...	...
Tuff	36	35	36	...	...
Obsidian	39	33	36	...	...
Calcined shale M	28	23	26	27	28
Calcined shale N	22	16	19	...	...
Calcined diatomaceous earth	32	26	29	...	...
Uncalcined diatomaceous earth	25	19	22	22	31
Synthetic silica glass	10	10	10	...	...

Effective admixtures should exhibit

some beneficial effect in addition to that of dilution. To determine the extent of this additional beneficial effect, the applicable data in Table V and VI were plotted in Fig. 3. The curves were interpolated (or extrapolated) to determine the percentage replacement that corresponded to the appropriate criteria for effectiveness. The interpolated values are shown in Table IX.

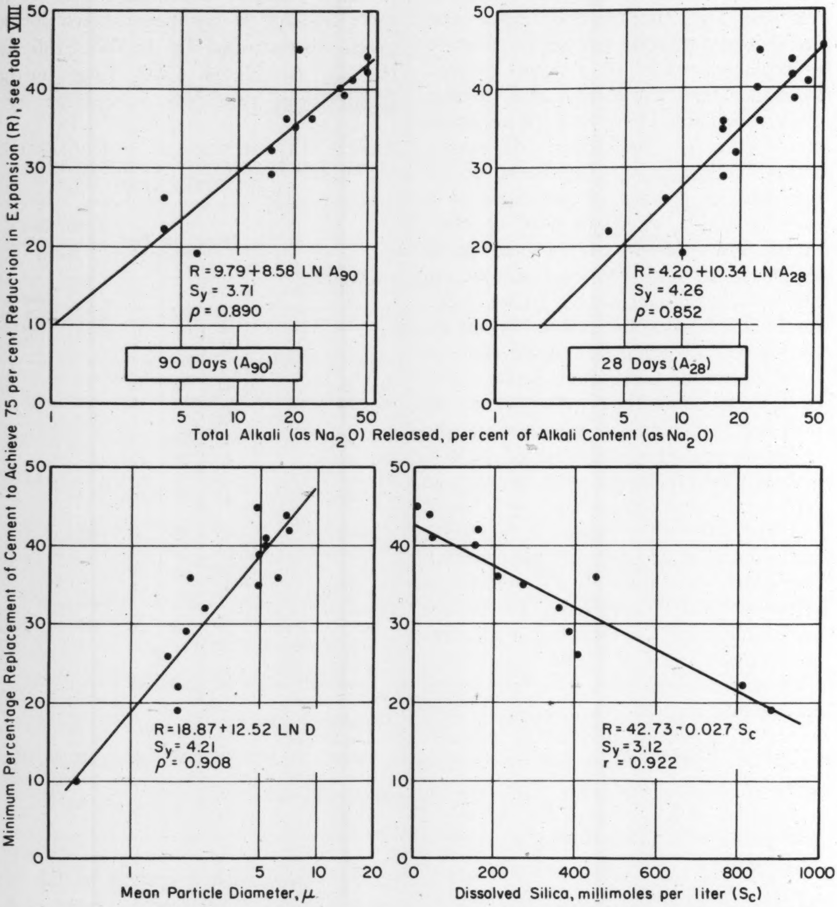


FIG. 4.—Correlation of Minimum Replacement Values with Alkali Release, Fineness, and Dissolved Silica.

All the admixtures tested do have some additional beneficial effect other than dilution of cement. Some effects are manifested at different levels of replacement ranging from a minimum of 10 per cent for synthetic silica glass at both 14 days and 6 months to 52 per cent for fly ash III at 14 days. The agreement between the results obtained for the two test methods is very good considering that these are interpolated results.

The minimum percentage replacement values may also be used to compare admixtures with each other. The lower the minimum replacement value, the more effectively the admixture will prevent expansion due to the alkali-aggregate reaction. This comparison is made in Table VIII. The admixtures are listed in ascending order of the average minimum replacement values. The average of the 14-day and 6-month values was used. The changes in the order would not be significant if either the 14-day or 6-month values were used. The synthetic silica glass heads the list as most effective, followed by the calcined shales and uncalcined diatomite, then the natural glasses, and finally the blast-furnace slags and fly ashes.

Correlation of Minimum Replacement Values with Fineness, Alkali Release, and Dissolved Silica:

The relation between the interpolated minimum replacement value for each admixture and the fineness of the admixture (mean particle diameter), the rate of alkali release at 28 and 90 days, and the amount of silica dissolved in the quick chemical test is indicated in Table VIII. The minimum replacement value did not correlate with the 0, 2, and 7-day alkali-release data. Figure 4 gives the curves of best fit, the equations of these curves, the correlation indices or coefficient, and the standard errors of estimate. The correlations, with due considerations of the small sample, are not due to random chance. Since evidence of correlation between the independent variables was not found, a multiple linear regression curve for the data in Table VIII was calculated with the following results:

$$R = 27.3 - 0.0158\,S_c + 1.6 \ln D + 3.5 \ln A_{90}$$

where: multiple correlation coefficient = 0.951, standard error of estimate = 2.50, and beta coefficient for S_c = 0.542, A_{90} = 0.366, D = 0.109.

The sample size is too small for a sound opinion to be formed regarding the multiple correlation, although it appears to be a good correlation. The beta coefficients indicate that the S_c value of an admixture has the greatest effect on the minimum percentage replacement value and the mean particle diameter the least. Other correlations were attempted including silica content, alkali content, alkali release as a percentage of the amount of admixture used, all *versus* the minimum replacement value, but without success. The correlations and also the lack of correlations indicate to some extent the factors that are necessary for effective reduction of expansion due to the alkali-aggregate reaction. Further work is necessary to determine the relative effect of lime on the reactions. The multiple correlation does not suggest the mechanism of the reaction but rather serves to evaluate the relative effect of the factors, when considered together, on the reaction.

Reactions Involved in Preventing Excessive Expansion:

Many hypotheses have been advanced to explain alkali-aggregate reaction and the effectiveness of measures to prevent its causing excessive expansion in concrete; however, a great deal of work still remains to be done to provide a basis for an adequate hypothesis. With the thought that some benefits might be derived from simplification of what appears to be a complex reaction, a summary of an hypothesis concerning the reactions involved was prepared, part of which has been published (1). It proposes a working hypothesis concerning phenomena involved in pozzolanic reaction, alkali-aggregate reaction, and prevention of excessive expansion of concrete due to alkali-aggregate reaction. The hypothesis was based on the assumption that all three phenomena are fundamentally the same: the reaction of calcium, alkalies, and hydroxide ions with soluble silica. The degree of reaction was thought to depend on the silica content, the metastability of the silica, and the fineness of the material. This investigation substantiates part of the working hypotheses. Since precise knowledge of the actual reactions that will cause the phenomena is lacking, the hypothesis will certainly aid in determining the type of chemical test needed to evaluate materials with respect to the phenomena. A chemical test to evaluate effectiveness of admixtures will have to determine, in the presence of lime, the reactive (glassy or noncrystalline) silica content of the material, and the alkalies that will be removed from solution. The chemical tests used in this investigation do not fulfill these requirements since:

(*a*) The quick chemical test does not evaluate the reductions in alkalies directly, nor does the reaction take place in the presence of lime.

(*b*) The modified quick chemical test does not correlate the effects of dissolved silica and reduction of alkalies with effectiveness in preventing excessive expansion. In addition, both tests lack the precision normally desired in chemical tests. The precision of the quick chemical test has been previously examined and reported (10).

CONCLUSIONS

All the materials tested in this study will effectively reduce expansion if sufficient amounts are employed in the mixture. All the materials exhibit a beneficial effect with regard to expansion other than by dilution. The synthetic silica glass was found to be the most active admixture, only 10 per cent replacement by volume is needed to reduce the percentage expansion by 75 per cent in two weeks; next the calcined shales, requiring 19 to 29 per cent replacement; the uncalcined diatomite, requiring 22 per cent; the volcanic glasses, requiring 32 to 36 per cent; and lastly, the slags and fly ashes, requiring 39 to 45 per cent.

It is evident from this investigation that neither chemical test can be used, with reliance, to evaluate the effectiveness of an admixture in preventing excessive expansion. Further work may develop a reliable chemical test, since a relation was found between minimum percentage replacement value, fineness of the material, reactive silica, and the amount of alkalies retained by the reaction product.

The mortar-bar test requires improvement to increase the precision for both intra- and interlaboratory testing. Andreasen and Christensen (3) have noted that the finer fractions (passing a No. 125 sieve) in the Pyrex aggregate may act as admixtures, and have stressed the importance of removing these finer fractions by washing the aggregate. Other differences in testing conditions and procedures between laboratories should be resolved.

Specifications for evaluating admixtures for effectiveness in preventing excessive expansion due to the alkali-aggregate reaction require the material to be tested at one percentage replacement, generally 25 per cent by volume. As indicated in this study, materials may be more effectively evaluated and compared if they are tested at two or three different percentage replacements. The minimum percentage replacement for effectiveness may then be determined by interpolation.

Pending additional development work, the criteria and procedures set forth in Specifications CRD-C 262 and

Methods 263 - 57[7] appear to be the most reasonable means of establishing the effectiveness of a material in preventing excessive expansion due to alkali-aggregate reaction.

Acknowledgments:

The work reported in this paper was conducted by the Concrete Division of the U. S. Army Engineer Waterways Experiment Station under the direction of Thomas B. Kennedy and under the supervision of Bryant Mather, J. M. Polatty, C. H. Willetts, R. V. Tye, R. L. Curry, Leonard Pepper, and Katharine Mather. The report on which this paper is based was prepared by Alan D. Buck, B. J. Houston, and Leonard Pepper. Valuable comments were provided by Raymond E. Davis, Director Emeritus, Engineering Materials Laboratory, University of California, Berkeley, Calif., Roy W. Carlson, consulting engineer, Berkeley, Calif., and R. L. Blaine, Chief, Concreting Materials Section, National Bureau of Standards, Washington, D. C.

REFERENCES

(1) American Concrete Institute Committee 212, Committee's Closure to Discussion of "Admixtures for Concrete." *Proceedings*, Journal Am. Concrete Inst., Vol. 51, December 1955, p. 148-6.

(2) Symposium on Use of Pozzolanic Materials in Mortars and Concrete, Am. Soc. Testing Mats. (1950). (Issued as separate publication *ASTM STP No. 99*.)

(3) A. H. M. Andreasen and K. E. Hauland Christensen, "Investigation of the Effect of Some Pozzolans on Alkali Reactions in Concrete," Progress Report L 1, Committee on Alkali Reactions in Concrete, Danish National Institute of Building Research and Academy of Technical Sciences, Copenhagen (1957).

(4) Mordecai Ezekiel, "Methods of Correlation Analysis," John Wiley and Sons, Inc., New York, N. Y., 2nd Edition, June 1947.

(5) P. E. Irick and J. B. Blackburn, "Application of Statistical Methods to Laboratory Concrete Freeze-Thaw Test Data," *Proceedings*, Highway Research Board, Vol. 34, pp. 329–367 (1955).

(6) Richard C. Mielenz, Kenneth T. Greene, Elton J. Benton, and Fred H. Geier, "Chemical Test for Alkali Reactivity of Pozzolans," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 1128 (1952).

(7) W. T. Moran, and J. L. Gilliland, "Summary of Methods for Determining Pozzolanic Activity," Symposium on Use of Pozzolanic Materials in Mortar and Concretes. Am. Soc. Testing Mats., p. 109 (1950). (Issued as a separate publication *ASTM STP No. 99*.)

(8) U. S. Bureau of Reclamation, "Calcined Reactive Siliceous Material for Use in Concrete," Specifications No. 1904, Appendix A to reference 7, Davis Dam Project, Denver, Colo. (1947)

(9) U. S. Army Engineer Waterways Experiment Station, CE, Handbook for Concrete and Cement (with quarterly supplements), Vicksburg, Miss., August 1949.

(10) Leonard Pepper, "Tests for Chemical Reactivity between Alkalies and Aggregates, Quick Chemical Test," Technical Memorandum No. 6-368, Report No. 1, Vicksburg, Miss., August 1953.

(11) Bryant Mather, "The Partial Replacement of Portland Cement in Concrete," Papers on Cement and Concrete, Am. Soc. Testing Mats., p. 37 (1958). (Issued as separate publication *ASTM STP No. 205*.)

---

[7] CRD-C 262-57, Corps of Engineers Specifications for Pozzolan as an Admixture for Use in Portland-Cement Concrete, and CRD-C 263-57, Corps of Engineers Methods for Sampling and Testing Pozzolan as an Admixture for Use in Portland-Cement Concrete, provide that, when pozzolan is to be used with aggregate which is regarded as potentially deleteriously reactive with the alkalies in cement, the pozzolan shall develop a reduction of mortar expansion at 14 days of at least 75 per cent when tested in mortar bars made with a cement containing at least 1 per cent total alkalies calculated as Na_2O and pyrex glass aggregate and a replacement of the cement by pozzolan in the amount of 25 per cent by absolute volume.

(12) R. V. Tye and B. Mather, "Tests for Chemical Reactivity between Alkalies and Aggregate, Mortar-Bar Test," CE, Technical Memorandum No. 6-368, U. S. Army Engineer Waterways Experiment Station, *Report No. 2*, Vicksburg, Miss., September 1956.

(13) B. Mather, "Investigation of Portland Blast-Furnace Slag Cements," Technical *Report No. 6-445*, U. S. Army Engineers Waterways Experiment Station, Vicksburg, Miss., Dec. 1956; *Journal of the American Concrete Institute* (*Proceedings*), Vol. 54, pp. 205–232 (1957).

DISCUSSION

MR. T. C. POWERS.[1]—I have not read this paper, hence this question. Do these correlations, pertaining to soluble silica, pertain to all the materials you described at first, or only to the selected four that you described later?

MR. BRYANT MATHER (*author*).—It pertained to the 14 of the 20 materials that belong to classes 1 and 3 through 6, as indicated by the values in Table VIII.

MR. A. D. CONROW.[2]—What is the authors's opinion of Pyrex glass as a material to represent a reactive aggregate and the reliability of results obtained with its use as a means for judging performance if common aggregates are used?

MR. MATHER.—I believe that Pyrex glass provides the most standardizable reactive aggregate to use as a representative of extreme reactivity. I do not envision the case where a mineral admixture would be effective in reducing expansion of a high-alkali cement - Pyrex glass mortar and fail to be effective in the field where the potential expansion was due to alkali-aggregate reaction.

There are expansions in the field, as is well known, which have not been proved to be due to alkali-aggregate reaction. Pyrex glass with high-alkali cement would not provide a controlled situation for testing for some other kind of expansion and the ability of something to prevent or reduce it.

MR. RAYMOND E. DAVIS.[3]—I am wondering if the results of the Pyrex glass test do not give an exaggerated idea of the quantity of pozzolan required to reduce the alkali aggregate expansion as compared with the results that we get with most of the reactive aggregates.

MR. MATHER.—I am sure that the results do tend to give such an exaggerated idea. It will be recalled that ASTM Specifications C 402 – 58 T provide alternative requirements for the ability of a mineral admixture to deal with alkali reaction expansion. The test with Pyrex glass and high-alkali cement and the requirement that a 75 per cent reduction in expansion be obtained at 14 days when 25 per cent by weight of the cement is replaced by the mineral admixture is the less desirable alternative. The preferable alternative is the test with Pyrex glass and job cement and the requirement that the 14-day expansion not exceed 0.020 per cent when the amount of admixture to be used on the job is used in the mortar. This latter approach not only allows the severity of the test to be reduced proportionately to the degree to which the job cement has less than a maximum alkali content but also permits the cement: admixture ratio to be that which will actually be employed. Whether we can and should also employ a less reactive aggregate than Pyrex glass is debatable. If we could be sure that the accelerated test gave in 14 days an expansion proportional to the ultimate expansion of the test combination in service and could also be sure that the test aggregate represented the most reactive condition to be encountered, then such a test aggregate—less reactive than Pyrex—would be desirable. Obviously an admixture should not be rejected for use because tests with a cement of 1.0 per cent alkalies as Na_2O, Pyrex glass, and a cement: admixture ratio of 4:1 showed less than 75 per cent reduction in expansion when it is certain that the cement will never contain more than say 0.7 per cent alkalies as Na_2O, the aggregates are only mildly reactive, and the cement: admixture ratio will be 3:1 rather than 4:1.

---

[1] Research Counselor, Portland Cement Assn., Skokie, Ill.

[2] Research Engineer, Ash Grove Lime and Portland Cement Co., Chanute, Kans.

[3] Director Emeritus, Engineering Materials Laboratory, University of California, Berkeley, Calif.

STUDIES OF LIMESTONE AGGREGATES BY FLUID-FLOW METHODS*

By W. L. Dolch[1]

Synopsis

Fluid-flow measurements were made in an attempt to learn more about the pore characteristics of coarse aggregates and their influence on the freezing-and-thawing durability of concrete. Determinations of density, porosity, absorption, degree of saturation, specific surface area, capillary absorptivity, permeability, and tortuosity factor were made on four Indiana limestones with both good and poor field and laboratory durability records. Absorptivity was found to be better than permeability as an index of surface area. The poor stones had consistently larger values of porosity, absorption, specific surface, permeability, absorptivity, ratio of absorptivity to permeability, and coefficient of rate of saturation increase than those for the more durable materials.

The performance of a rock as a concrete coarse aggregate is determined to a large extent by the properties of its internal void system. Rhoades and Mielenz (**1**)[2] state "In a broad sense, the internal pore characteristics of the particles are the most important properties of the aggregate materials." This influence is primarily due to the effect on the absorption and retention of water by the aggregate. The details of this influence are imperfectly known and current standard test methods reflect this imperfection. Such methods involve the determination of simple properties of the pore system and leave much to be desired from the standpoint of reliability in performance prediction.

The properties of aggregates, and specifically the influence of pore structure on the durability of concrete, have been reviewed extensively (**1,2,3,4,5**). An important aspect is the influence of the porosity of the aggregate on the durability of concrete in freezing and thawing. A discussion of the theoretical aspects of frost action has been given recently by Powers (**6**). He points out that damage caused by water freezing in the aggregate pieces can be brought about in several different ways. Perhaps, chief among these is rupture of the surrounding matrix or of the aggregate piece itself by the hydraulic pressure generated when the growing ice forces water through the porous material. The pressure established is proportional to the rate of movement of the water and to the distance that it must traverse and is inversely proportional to the permeability of the medium. For every material and condi-

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Research Associate, Joint Highway Research Project, Purdue University, Lafayette, Ind.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

tion of freezing there is a "critical size" that is the length of flow path that cannot be exceeded without the generation of stresses harmful to the material.

Inherent in such a mechanism are questions of the acquisition and retention of water by the material and of the degree to which it opposes internal movement of water—that is, its permeability.

These properties of capillarity and permeability can also give an indication of the pore structure of a porous medium. In moving through a material a liquid experiences viscous drag that is a function of the size of the pores through which it is traveling. In absorption the force causing ingress of the liquid is a capillary pressure that is related to the size of the channel in question. The relationship between these properties and pore size is, however, capable of analytical expression only for simple pore structures. The pore systems of the rocks in question here are so complicated that it is necessary to postulate a model that has the same phenomenological properties as the substance it represents but has a pore system that is simple enough so that analytical relationships that relate its structure to the measured properties can be derived. Much work has been done on a variety of materials; for details of this work and the theory supporting it the reader is referred to the texts by Carman (**7**) and Scheidegger (**8**) and to various reviews (**9,10,11,12**).

The approach used in the work described here was the measurement of the permeability and capillary absorptivity, along with various associated properties, of four Indiana limestone aggregates, two of which have good and two poor performance histories in pavements. These experimental results were used to compute the properties of the pore system of a model, and tentative correlations are indicated between these properties and the durability histories of the materials.

EXPERIMENTAL WORK

Test Materials:

The materials used were four limestone rocks that have well-documented histories in Indiana. These materials have been used for several other studies. These previous studies (**13,14,15,16,17,18,19**) have shown that distress of Indiana pavements due to blowups and frost action is traceable to the coarse aggregate component of the concrete. The aggregates with poor performance usually have larger values of absorption, porosity, and vacuum degree of saturation than do those with good performance. The poor materials also have a comparatively large porosity with a "pore diameter" smaller than 4 μ. These poor-performing materials have also been shown to be generally free from alkali reactivity and from thermal incompatibility. The conventional tests for frost susceptibility are sometimes inadequate when applied to these materials, but field performance is duplicated, in a relative sense, by the results of freezing-and-thawing tests of concrete.

TABLE I.—COMPOSITION AND DURABILITY PERFORMANCE OF AGGREGATES.

Source	Calcite, per cent by weight	Dolomite, per cent by weight	Field Performance	Approximately 100-Cycle Durability Factor (**18,19**)
No. 1-1S.......	3	93	good	98
No. 9-1S.......	5	89	poor	35
No. 47-2S.......	77	17	poor	40
No. 67-2S.......	97	2	good	98

The quarries were sampled. No statistical precautions were taken; the samples were merely picked with an eye toward similarity to the general mass and freedom from obvious anomalies. Although the quarries are comparatively homogeneous, this sampling procedure does necessitate the specific assumption that if pavements were made using the samples as aggregates they would have durability histories similar to those of the pavements that were actually made with material from the same sources. This sampling procedure was felt to be adequate for an exploratory study.

In Table I are listed the pavement performance histories of the materials, their calculated compositions, and the approximate 100-cycle durability factors[3] (18,19) of concrete made with the vacuum-saturated aggregates and having approximately 3.5 per cent air contents.

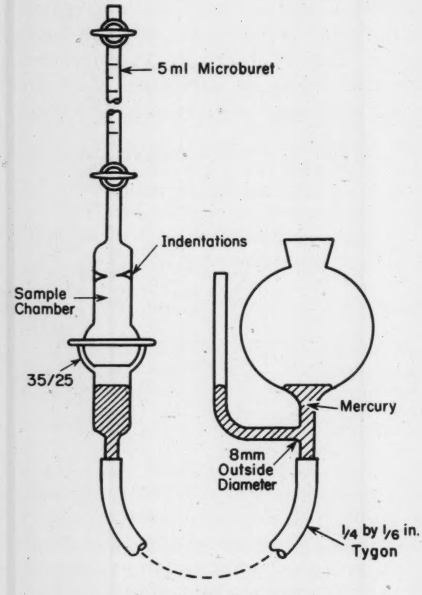


FIG. 1.—Porosimeter.

Sample Preparation:

Small cores, approximately 1.7 cm in diameter and several centimeters long, were cut from the samples. The cores so obtained were then faced off and the ends were ground by hand to be parallel and normal to the cylindrical axis. In other instances the test pieces were prepared by sawing out small prisms of rock and hand-finishing the surfaces.

Density and Absorption:

The values for the bulk densities were obtained by measurement of linear dimensions and weighing. Values for 24-hr absorption were obtained in the usual way. The samples were then dried and vacuum-saturated by evacuating for 1 hr with an aspirator and allowing to soak an additional 23 hr. They were then surface-dried, weighed, and the vacuum absorptions were calculated.

Porosity and Degree of Saturation:

The porosity, ϵ, is defined as the ratio of the pore volume to the bulk volume. The porosities of the samples were determined by means of an adaptation of the "McLeod Gage" porosimeter (Fig. 1) of Washburn and Bunting (20). The porosity was measured by raising the mercury until it covered the sample in the chamber and trapped the air in the pores. The level was raised to the top mark of the microburet and the upper stopcock was closed. The mercury was then lowered to expose the sample. The air came out of the pores and was trapped when the mercury level was again raised. The volume of air, which equals the volume of pores, was read directly from the microburet. The degree of saturation, S_w, or, more simply, the saturation is the ratio of the volume of water in the pores to the total pore volume. The 24-hr and vacuum saturations were calculated from the data obtained in the absorption and porosity determinations.

---

[3] See ASTM Method C 291 - 57 T, Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water, 1958 Book of ASTM Standards, Part 4, p. 688, for a definition of the durability factor. A high factor indicates relatively durable performance in laboratory freezing-and-thawing tests.

TABLE II.—PHYSICAL PROPERTIES OF AGGREGATES.

Source	Bulk Density, g per cu cm	Porosity, per cent	Absorption, per cent		Saturation, per cent	
			24-hr	Vacuum	24-hr	Vacuum
No. 1-1S	2.73	2.31	0.72	1.17	58	89
No. 9-1S	2.20	24.20	7.58	10.28	69	93
No. 47-2S	2.42	10.30	2.51	3.75	59	87
No. 67-2S	2.67	2.46	0.55	0.61	59	67

In Table II are listed the bulk density, porosity, absorption and saturation values obtained. Each value is an average of the results of measurements on five to ten separate samples.

Permeability:

The permeability of a porous medium is defined by the expression

$$\frac{V}{tA} = \frac{K\Delta P}{\eta L} \qquad (1)$$

where:

V = the volume of permeant traversing the sample under steady-state conditions in time t,

A = the gross area of the sample normal to the flow direction,

ΔP = the pressure difference causing flow,

η = the viscosity of the permeant,

L = the gross length of the sample, and

K = the permeability.

The cgs units for K are cm^2. This unit is so large that another has come into general use. This is the millidarcy (md) and 1 cm^2 equals 1.013×10^{11} md.

The apparatus used to determine the permeabilities is shown in Fig. 2. The dry sample was mounted in the holder with a hard asphalt cement. The assembled apparatus was pumped down to the higher pressure desired. The bypass stopcock was closed and the pumping was continued until the desired pressure difference was established across the sample. The system was then closed and the decay of the pressure differential was measured with a cathetometer as the air flowed through the sample. A knowledge of the rate of pressure change, the sample's dimensions, and certain apparatus constants permitted the calculation of the permeability of the sample. The apparatus is of the same principle as those used by Arnell (22) and Kamack (23).

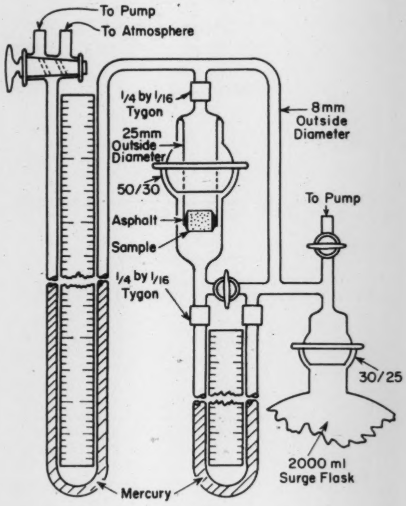


FIG. 2.—Permeameter.

When a gas is used as the permeant there are two restrictions on the definition of permeability (Eq 1). These are (1) the volume, V is that corresponding to the mean pressure across the sample and (2) K is a function of the mean pressure. This second restriction is due to the fact

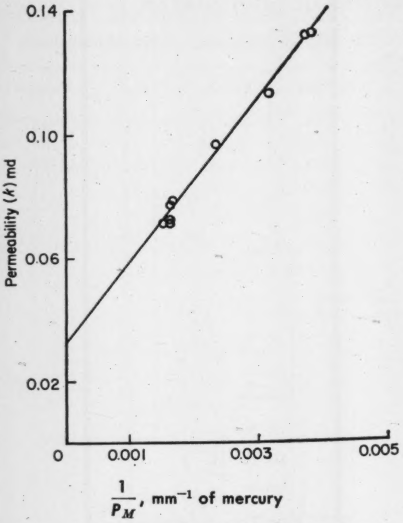


FIG. 3.—Permeability of No. 47-2S-P-98.

TABLE III.—FLUID-FLOW PROPERTIES OF AGGREGATES.

Source	Permeability, md	Absorptivity, sq cm per sec	Tortuosity, k_t	Tortuosity at 24-hr Saturation, k_{ta}
No. 1-1S	0.00205	4.59×10^{-9}	145.	430.
No. 9-1S	4.30	3.24×10^{-5}	4.51	9.52
No. 47-2S	0.032	5.00×10^{-7}	29.9	85.0
No. 67-2S	0.0072	6.18×10^{-9}	29.3	83.8

that at pressures at which the mean free path of the gas is roughly equal to the size of the flow channel a gas starts to "slip" past the channel walls and the apparent permeability is enhanced. The influence of pressure is shown by the equation of Klinkenberg (21),

$$K = K_l\left(1 + \frac{b}{P_m}\right) \quad \text{.........} (2)$$

where P_m is the mean pressure across the sample. Klinkenberg showed that the intercept K_l is the permeability that would be obtained for the flow of a liquid through the sample. The permeabilities of these samples were, therefore, measured at several mean pressures and plotted against reciprocal mean pressure. An example of the results is shown in Fig. 3. The values of K_l, the intercept, obtained in this way are listed in Table III. They are for only one sample from each source, but this sample was carefully selected to be as representative of the group as possible.

Absorptivity:

When a liquid is absorbed into a solid by capillary action the volume absorbed at any time is proportional to the approach area of the sample and to the square root of the time elapsed. Powers and Brownyard (24) defined the "absorptivity," K_a by

$$\left(\frac{V}{A}\right)^2 = K_a t \quad \text{............} (3)$$

where V is the volume of capillary permeant taken up in time t through approach area A. The cgs units on K_a are cm^2 per sec.

The absorptivity was measured in a simple, yet apparently precise and accurate, way. The sample was taped with plastic tape to prevent evaporation of water from the sides. It was then placed on several filter papers that were wet with water and a stop watch was started. From time to time the watch was interrupted and the sample was removed and weighed rapidly. It was then returned to the (essentially) free water surface and timing was resumed. It is assumed that a negligible amount of flow took place within the sample during the weighing process.

The logarithm of V/A was plotted against the logarithm of t and K_a was calculated from the 1-min intercept. In most runs only enough points were taken to define the line. But for two samples from each source the process was allowed to continue until equilibrium was reached in order to determine the degree of saturation. With the denser rocks it took several days to complete such a run. The filter paper was kept moist by means of a wick and water supply.

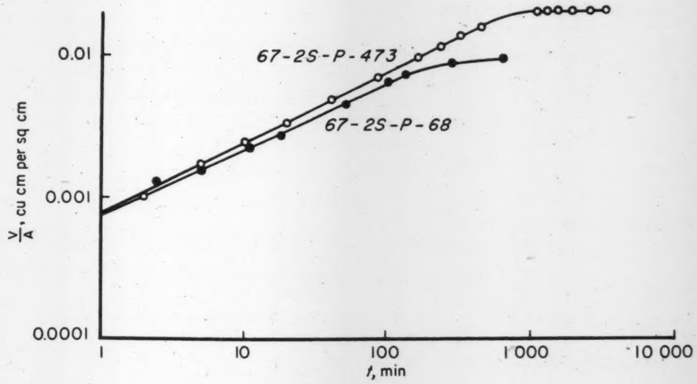


FIG. 4.—Absorptivity of No. 67-2S.

An example of these curves is given in Fig. 4. The values obtained for K_a are listed in Table III and are averages for seven individual samples except for No. 1-1S for which only four were used.

The values for the capillary saturation obtained in this way were so close to the 24-hr immersion values that the latter were used in subsequent calculations.

Tortuosity:

It is reasonable to suppose the path of a "particle" of fluid flowing through a porous medium to be longer than the length of the specimen between inlet and outlet faces. The square of the ratio of this tortuous length of flow, L_t, to the gross length, L, is called the tortuosity, k_t, of the pore system. The method used to measure the tortuosities was discussed by Wyllie and Spangler (25) and depends on the assumption of average equality between the path traversed by a fluid flowing in a porous medium and that traversed by an electrical current when the pores contain a conducting solution. The ratio of the electrical resistance of the medium containing the electrolyte to that of an amount of solution with the same gross dimensions as the sample is called the formation factor, F. If the pore space is not completely saturated with electrolyte, the formation factor, F_e, will be larger than F. The ratio of F_e to F is called the resistivity index, I, and this has been found to be a simple function of the degree of saturation,

$$\frac{F_e}{F} = I = S_w^{-n} \dots\dots\dots\dots (4)$$

The exponent n seems to be independent of the saturation until comparatively low saturations are reached. Measurements on a variety of porous media have shown n to be equal to $2 \pm \frac{1}{2}$. Analysis of the problem leads to an expression for tortuosity in terms of the formation factor and the porosity,

$$k_t = (F\epsilon)^2 \dots\dots\dots\dots (5)$$

The two ends of the sample prisms were coated with conducting paint. The samples were then vacuum-saturated with a 5 per cent sodium chloride solution. They were blotted surface dry, weighed rapidly, and their resistances were measured with a 2000-cycle bridge using an oscilloscope as a null indicator. The exponent n in Eq 4 was taken to be 2. These data permitted the calculation of the tortuosity by means of Eqs 4 and 5.

TABLE IV.—SPECIFIC SURFACES, SQ M PER G.

Source	Sorption	Permeability	Absorptivity
No. 1-1S........	0.67	0.046	0.46
No. 9-1S........	2.04	0.238	5.85
No. 47-2S........	2.54	0.271	2.22
No. 67-2S........	1.09	0.061	2.22

TABLE V.—PORE RADII,[a] μ.

Source	Sorption	Permeability	Absorptivity
No. 1-1S........	0.03	0.32	0.03
No. 9-1S........	0.11	0.79	0.03
No. 47-2S........	0.03	0.23	0.03
No. 67-2S........	0.02	0.26	0.01

[a] The values in this table have been rounded to two decimal places to facilitate comparisons.

Because of uncertainties with respect to various aspects of this determination, including those inherent in the basic analogy on which the method depends, the tortuosity values reported in Table III are the most unreliable data reported here. Also, only one sample from each source was used to obtain these values.

If the pore space is only partially saturated, the tortuosity of the liquid phase will be higher than k_t. The column in Table III headed "Tortuosity at 24-hr saturation" gives the values calculated by means of

$$k_{ta} = k_t S_w^{2(1-n)} \qquad (6)$$

where k_{ta} is the tortuosity considered to be applicable to the water phase during capillary absorption, that is, with the 24-hr saturation values used for S_w and n equal to 2.

Water Vapor Adsorption:

The specific surface areas of the rocks were measured by means of the water vapor adsorption technique and the theory of Brunauer, Emmett, and Teller (26). The isotherms were determined using 16 to 30-mesh samples and the ordinary gravimetric technique with a vacuum desiccator and saturated salt solutions for the establishment of constant vapor pressure. The sorptions were measured up to approximately 30 per cent humidity. The values obtained for the specific surfaces of the samples are listed in Table IV.

Discussion

The relationship between the permeability and pore characteristics that will be used here is the Kozeny-Carman equation. This relationship involves a model that has two attributes of most real porous media, a tortuous length of flow path and an irregular cross-section of the flow channels. The derivation of the equation is given by Carman (7,10) and the result is

$$K_l = \frac{\epsilon m^2}{k_o k_t} \qquad (7)$$

where k_o is a factor related to the cross-sectional shape of the pores and m is the hydraulic radius of the pores, that is, the ratio of the cross-sectional area to the perimeter. By means of the relationship $m = \epsilon / S_g \rho_B$, where ρ_B is the bulk density of the porous solid and S_g is its specific surface, that is, the surface area of 1 g of the solid, Eq 7 can be transformed to:

$$K_l = \frac{\epsilon^3}{k_o k_t S_g^2 \rho_B^2} \qquad (8)$$

The shape factor, k_o, is equal to 2 for a round cross-section and changes only a little for other shapes. For consolidated porous media, Wyllie and Spangler (**25**) gave evidence in favor of 2.7, and that value is used here. For a model of round tubes of radius r, Eq 7 becomes:

$$K_l = \frac{\epsilon r^2}{8k_t} \ldots\ldots\ldots\ldots (9)$$

The values of specific surface, calculated by means of Eq 8, are given in Table IV, and those for the radii, calculated from Eq 9, are given in Table V both under the heading "Permeability."

When water enters a capillary tube and progresses through it, the equation describing the flow can be simplified by the assumption that the only forces of consequence are the capillary force causing ingress of the water and the viscous drag that retards permeation. With the further assumption that the contact angle of water on these rock surfaces is zero degrees, the final result of equating these forces for dynamic equilibrium is

$$\gamma m = k_o \eta x \left(\frac{dx}{dt}\right) \ldots\ldots\ldots (10)$$

where x is the tortuous distance the liquid has progressed in time t, η is the viscosity and γ the surface tension of the liquid, and the other symbols are as previously defined. If the area of the tube normal to the tortuous flow direction is a, the volume of liquid in it at time t will be ax and the rate of change of this volume will be

$$\frac{dV}{dt} = \frac{adx}{dt} \ldots\ldots\ldots\ldots (11)$$

If the tube is considered to consist of the entire interconnected pore space, then the fraction of the bulk volume that is penetrated by means of capillarity is defined as the capillary porosity, α, that is,

$$\alpha = \frac{aL_t}{AL} = \frac{ak_{t\alpha}^{1/2}}{A} \ldots\ldots\ldots (12)$$

Elimination of x and a from Eqs 10, 11, and 12 gives

$$\frac{VdV}{dt} = \frac{\gamma m A^2 \alpha^2}{k_o \eta k_{t\alpha}} \ldots\ldots\ldots (13)$$

When this equation is solved, the initial condition of $V = 0$ for $t = 0$ is imposed, and the definition of K_a (Eq 3) is inserted, the final result is

$$K_a = \frac{2\gamma m \alpha^2}{k_o k_{t\alpha} \eta} \ldots\ldots\ldots (14)$$

which becomes, for round tubes of radius r,

$$K_a = \frac{r\gamma\alpha^2}{2\eta k_{t\alpha}} \ldots\ldots\ldots (15)$$

If, finally, it is postulated that the hydraulic radius for the noncapillary-penetrable pore space is equal to that for the capillary pore space, then $\rho_B S_g m = \epsilon$ and elimination of m from Eq 14 gives

$$S_g = \frac{2\gamma\epsilon\alpha^2}{k_o k_{t\alpha} K_a \rho_B \eta} \ldots\ldots\ldots (16)$$

The values for specific surface calculated from Eq 16 are given in Table IV and those for the radii calculated from Eq 15 are given in Table V, both under the heading of "Absorptivity."

The column in Table V labelled "Sorption" is the radius of round pores that would have a specific surface equal to that measured in the water vapor sorption determinations.

A limitation of the Kozeny equation is that the pore size should be reasonably uniform since a few large pores contribute to the permeability all out of proportion to their surface area. Tables IV and V show this effect. The radii calculated from permeability are all comparatively large and the specific surfaces are small, indicating the reflection of the larger pore size by this method of measurement. The same situation has been described by others (**7,27**).

On the other hand, the absorptivities should give a smaller pore size and a larger surface area because the liquid will tend to accumulate in these smaller pores as a result of their higher capillary potential. Tables IV and V seem to show this effect also. The calculated radii are smaller and the surfaces larger than are the values derived from the permeabilities. The values presented can be considered only as indications of the trend and should probably not be taken too literally because of the inaccuracies of the tortuosity determinations, among other things. This is amply evidenced by two of the four absorptivity surface values being higher than the sorption surface, which is the theoretical maximum. The trend, however, seems to be there and the absorptivity method is probably better than the permeability method as an interpretation of specific surface—at least for the kind of materials considered here.

TABLE VI.

Source	K_a:2 ϵ^2, sq cm per sec	K_a:K_1 , sec^{-1}
No. 1-1S.......	0.4×10^{-5}	2.3×10^5
No. 9-1S.......	27.6	7.6
No. 47-2S.......	2.4	15.8
No. 67-2S.......	0.5	0.9

With respect to problems of frost action in aggregate particles, the data in Tables IV and V show no obvious distinctions between the good and bad materials. Blaine, Hunt, and Tomes (**28**) concluded that materials with poor frost durability have comparatively high specific surfaces, and the data reported here lend some support to this conclusion.

Inspection of Table II shows the poor materials to have higher porosities and absorptions than do the good ones. It also shows that a good material usually has a low value of vacuum saturation, while a high value, especially one greater than about 91 per cent, means a poor material, but that a border line region exists where the distinction cannot be made on this basis. All this, however, is nothing new and has been reported before.

The permeability and absorptivity values in Table III show order of magnitude differences between the good and poor stones. These differences may be indicative but, on the other hand, they are what would be expected for materials whose porosities have the variations of these.

Two new parameters have been developed that seem to distinguish between the good and poor materials and have, furthermore, a logical support from the theoretical concepts of frost action.

Powers (**6**) states "a principal difference among different kinds of rock particles is the rate at which they become saturated when given free access to water." Wray and Lichtefeld (**29**) also showed the importance of this characteristic. A simple derivation shows that the time rate of change of the saturation can be expressed as

$$\frac{dS_w}{dt} = \frac{K_a}{2\epsilon^2}\left(\frac{1}{S_w}\right) \quad \text{.........(17)}$$

Values of $K_a/2\epsilon^2$ for these materials are given in Table VI and do show a separation between the good and poor aggregates. Indeed, No. 9-1S, is given the stigma that it deserves from its field history.

The ratio of K_a to K_l should, logically, be a measure of the susceptibility of a particle to frost action. A high absorptivity means a comparatively high rate of absorption while a low permeability means a comparative reluctance of the material to permit the passage of water and, therefore, a smaller critical size. Both effects should tend toward an increased frost susceptibility and so the higher the ratio the worse. Similarly a rock with bulges in the pore system that can act to improve durability in the same way as those in an air-entrained cement paste do should show a comparatively low value of this ratio. These ratios are given in Table VI and do show the separation in the logical direction between the good and poor materials.

CONCLUSIONS

Based on the materials studied and the tests performed the following conclusions seem reasonable:

1. The rate of increase of the degree of saturation of a material when exposed to free water, as measured by the ratio of $K_a : 2\epsilon^2$, and the ratio of the absorptivity to the permeability are two indices of frost susceptibility when the material is used as a concrete coarse aggregate. The more susceptible materials can be expected to have comparatively large values of both these ratios.

2. The absorptivity is superior to the permeability as an index of surface area of a porous solid when a simple model is used to analyze the results.

Acknowledgments:

This work was financed by the State Highway Department of Indiana through the Advisory Board of the Joint Highway Research Project. This support is gratefully acknowledged.

REFERENCES

(1) R. Rhodes and R. C. Mielenz, "Petrographic and Mineralogic Characteristics of Aggregates," Symposium on Mineral Aggregates, Am. Soc. Testing Mats., p. 20 (1948). (Issued as separate publication *ASTM STP No. 83.*)

(2) D. W. Lewis, W. L. Dolch, and K. B. Woods, "Porosity Determinations and the Significance of Pore Characteristics of Aggregates," *Proceedings,* Am. Soc. Testing Mats., Vol. 53, 949 (1953).

(3) D. W. Lewis and W. L. Dolch, "Porosity and Absorption," Symposium on Significance of Tests of Concrete, Am. Soc. Testing Mats., (1955). (Issued as separate publication *ASTM STP No. 169.*)

(4) F. E. Jones, "The Physical Structure of Cement Products and Its Effect on Durability," *Proceedings,* Third Int. Symposium on the Chemistry of Cement (London), p. 368 (1952).

(5) R. F. Blanks, "Modern Concepts Applied to Concrete Aggregates," *Proceedings,* Am. Soc. Civil Engrs., *Vol.* 75, p. 441 (1949).

(6) T. C. Powers, "Basic Considerations Pertaining to Freezing-and-Thawing Tests," *Proceedings,* Am. Soc. Testing Mats., Vol. 55, p. 1132 (1955).

(7) P. C. Carman, "Flow of Gases Through Porous Media," Academic Press Inc., New York, N. Y. (1956).

(8) A. E. Scheidegger, "The Physics of Flow Through Porous Media," Macmillan Co., New York, N. Y. (1957).

(9) N. K. Adam, "Principles of Penetration of Liquids Into Solids," *Discussions Faraday Soc. No. 3,* p. 5 (1948).

(10) P. C. Carman, "Some Physical Aspects of Water Flow in Porous Media," *Discussions Faraday Soc. No. 3,* p. 72 (1948).

(11) P. C. Carman, "Properties of Capillary-Held Liquids," *Journal of Physical Chemistry,* Vol. 57, p. 56 (1953).

(12) R. M. Barrer, "Fluid Flow in Porous Media," *Discussions Faraday Soc. No. 3,* p. 61 (1948).

(13) K. B. Woods, H. S. Sweet and T. E. Shelburne, "Pavement Blowups Correlated with Source of Coarse Aggregate," *Proceedings,* Highway Research Board, Vol. 25, p. 147 (1946).

(14) F. O. Slate, "Chemical Reactions of Indiana Aggregate in Disintegration of Concrete," *Proceedings,* Am. Soc. Testing Mats., Vol. 49, p. 954 (1949).

(15) H. S. Sweet, "Research on Concrete Durability as Affected by Coarse Aggregates," *Proceedings,* Am. Soc. Testing Mats., Vol. 48, p. 988 (1948).

(16) N. Y. Lu, "Comparison of the Thermal and Moisture Expansion of Concrete and Stone," Thesis, Purdue University, Lafayette, Ind. (1947).

(17) D. W. Lewis and K. B. Woods, "Research as Related to the Development of Aggregate Specifications," *Reprint No. 51,* Purdue University, Engineering Experimental Station (1950).

(18) S. L. Bugg, "Effect of Air Entrainment on the Durability Characteristics of Concrete

Aggregates," *Proceedings*, Highway Research Board, Vol. 27, p. 156 (1947).

(19) J. B. Blackburn, "Freeze-and-Thaw Durability of Air-Entrained Concrete Using Indiana Aggregates," *Proceedings*, Highway Research Board, Vol. 28, p. 171 (1948).

(20) E. W. Washburn and E. N. Bunting, "Porosity. VII. The Determination of the Porosity of Highly Vitrified Bodies," *Journal*, Am. Ceramic Soc., Vol. 5, p. 527 (1922).

(21) L. J. Klinkenberg, "The Permeability of Porous Media to Liquids and Gases," Am. Petroleum Inst., Drilling and Production Practice, p. 200 (1941).

(22) J. C. Arnell, "Permeability Studies. II. Surface Area Measurements of Inorganic Pigment Powders," *Canadian Journal of Research*, A 25, p. 191 (1947).

(23) H. J. Kamack, "Simple Air-Permeability Method for Measuring Surface Areas of Fine Powders," *Analytical Chemistry*, Vol. 26, p. 1623 (1954).

(24) T. C. Powers and T. L. Brownyard, "Studies of the Physical Properties of Hardened Portland Cement Paste," *Proceedings*, Am. Concrete Inst., Vol. 43, p. 865 (1947).

(25) M. R. J. Wyllie and M. B. Spangler, "Application of Electrical Resistivity Measurements to Problems of Fluid Flow in Porous Media," *Bulletin*, Am. Assn. Petroleum Geologists, Vol. 36, p. 359 (1952).

(26) S. Brunauer, P. H. Emmett, and E. Teller, "Adsorption of Gases in Multimolecular Layers," *Journal*, Am. Chemical Soc., Vol. 60, p. 309 (1938).

(27) S. Brooks and W. R. Purcell, "Surface Area Measurements on Sedimentary Rocks," *Journal of Petroleum Technology*, Vol. 4, p. 289 (1952).

(28) R. L. Blaine, C. M. Hunt, and L. A. Tomes, "Use of Internal Surface Area Measurements in Research on Freezing-and-Thawing of Materials," *Proceedings*, Highway Research Board, Vol. 32, p. 298 (1953).

(29) F. N. Wray and H. J. Lichtefeld, "The Influence of Test Methods on Moisture Absorption and Resistance of Coarse Aggregate to Freezing-and-Thawing," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1007 (1940).

DISCUSSION

CHAIRMAN E. W. CUMMINS.[1]—I should like to ask one question. Is it possible, with further work along this line, that a test might be developed for acceptance or rejection of limestone for use in concrete?

MR. W. L. DOLCH (*author*).—Naturally, that is the end point of any study such as this, but it must be emphasized that these results apply only to similar materials. Whether you could take granite, for instance, and get the same results, I do not know.

MR. BRYANT MATHER.[2]—Approaches have been made in routine testing to things that are probably related to these parameters. I have reference particularly to some of the ratio procedures that have been in the literature on building stones for many years where, by some standardized procedure, you merely measure the increasing degree of saturation through a treatment of the samples by various saturating processes. These were reviewed by Schaffer in 1932.[3]

From the chairman's question about reducing this sort of thing to a test method, what people who are confronted with that problem will want to do when they get to it, is make it simple. Would it be expected that information of value for that purpose might be obtained if studies such as the author's included the determination of these simple parameters and perhaps at least some empirical relation might be found?

---

[1] Engineer in Charge of Aggregate Section, Ohio State Highway Testing Laboratory, Ohio State University, Columbus, Ohio.

[2] Engineer (Concrete Research), Concrete Division, Waterways Experiment Station, Jackson, Miss.

[3] R. J. Schaffer, "The Weathering of Natural Building Stones," D.S.I.R. Building Research, Special Report No. 18, His Majesty's Stationery Office, London (England), pp. 49–50 ("Saturation Coefficient") (1932).

MR. DOLCH.—We probably should have done it. With respect to simplicity of tests, I will point out that the measurement of the absorptivity is an extremely simple process once you have a shaped specimen. The measurement of porosity is a simple process and therefore the first of the two ratios mentioned in the paper could be determined easily. Permeability is not so easily measured but it is not hard either and so it is conceivable that studies of this sort, if the approach proves to be of value, should not be too difficult to transfer into standard test methods.

MR. CHARLES VOLLICK.[4]—I would like to ask the author whether he has made any attempt to correlate some of the standard tests such as sodium sulfate or magnesium sulfate soundness, with the values he has obtained and how they correlate with freezing-and-thawing data on concrete specimens.

MR. DOLCH.—These tests have been made on all these materials by others. I did not compare the results with these data. But for instance, this very poor material (No. 9–1S) with a 7½ per cent absorption will pass the sodium sulfate test in Indiana.

The standard acceptance tests are not invariably good indications of the durability of these materials.

---

[4] Chief, Engineering Laboratories, SIKA Chemical Corp., Passaic, N. J.

EFFECT OF LENGTH TO DIAMETER RATIO ON COMPRESSIVE STRENGTH—AN ASTM COOPERATIVE INVESTIGATION*

By Clyde E. Kesler[1]

Synopsis

Ten laboratories participated in a program planned by Subcommittee IIIa, Methods of Testing Concrete for Strength of ASTM Committee C-9 on Concrete and Concrete Aggregates, to determine the effect of the length to diameter ratio on the apparent compressive strength of concrete. The concrete strength, type of aggregate, specimen size, and curing were variables. At present, ASTM provides only one set of factors for "correcting" strengths obtained from specimens which have a length to diameter ratio less than the standard, 2. The cooperative study clearly shows that the correction factors are dependent on strength and also that lightweight aggregates require different factors than do normal-weight concretes.

The standard specimen for determining the strength of concrete is a 6 by 12-in. molded cylinder; however, it is not always possible to obtain a specimen of these exact dimensions. Specimens of different dimensions, unfortunately, yield different unit strengths for the same quality concrete. For a given diameter, but different lengths of specimens, the apparent strength will vary as shown in Fig. 1. There is little change in strength for length to diameter ratios, *l/d*, from 1.5 to 2.5, and any correction needed is small and not very important. However, below 1.5 there is a significant increase in the correction needed.

Previous studies by Murdock and Kesler[2] and by the Union of Testing and Research Laboratories for Materials and Structures[3] (RILEM) have shown that strength itself is an important variable in correcting the apparent strength of specimens with low *l/d* ratios. The present factors in ASTM Standard Methods of Securing, Preparing and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 – 57)[4] involve the variation of the *l/d* ratio and no other variable. As a result the corrected strengths of the weaker concretes may be higher than the probable strength of the standard cylinder. The higher strength concretes are penalized by the present correction factors.

The purpose of this cooperative investigation was to determine the effects of strength, aggregate, diameter, air entrainment, and curing on correction factors for various *l/d* ratios. This paper is a brief summary of the more apparent

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] John W. Murdock and Clyde E. Kesler, "Effect of Length of Diameter Ratio of Specimen on the Apparent Compressive Strength of Concrete," ASTM Bulletin, No. 221, April 1957, pp. 68–73 (TP 76).

[3] "Correlation Factors Between the Strengths of Different Specimen Types," *Bulletin 17*, Union of Testing and Research Laboratories for Materials and Structures, Paris, pp. 81–105.

[4] 1958 Book of ASTM Standards, Part 4, p. 706.

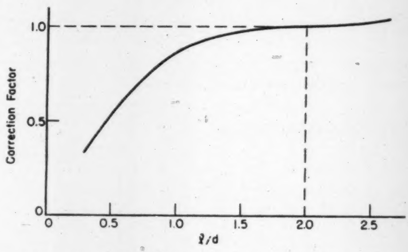


FIG. 1.—Typical Variation of Correction Factor With l/d Ratios.

results and does not represent an exhaustive analysis of the data.

ORGANIZATION OF THE PROGRAM

The program was sponsored by Subcommittee III a on Methods of Testing Concrete for Strength, of ASTM Committee C-9 on Concrete and Concrete Aggregates. Subcommittee III a has the responsibility for ASTM Methods C 42.[4]

Each of the ten cooperating laboratories was requested to make tests involving one or two important variables in addition to l/d ratio. In all cases an attempt was made to plan the program in such a way that each laboratory would use materials and equipment which were immediately available and with which the laboratory was familiar. After the tests were completed, the results were returned to the author for study. The program was sufficiently complicated and so much data was produced that all facets of the study cannot be considered in the limited space available for this paper. Nevertheless, the more important ones are presented and discussed.

The laboratories are identified by cap-

TABLE I.—STRENGTH CORRECTION FACTORS FOR 1000-psi CONCRETE.

Cooperating Laboratories	Aggregate	Slump, in.	Age, days	l/d Ratio					
				0.50	0.75	1.00	1.50	2.00	2.50
MOLDED CYLINDERS—6-IN. DIAMETER (UNLESS NOTED OTHERWISE)									
NORMAL WEIGHT—NON AIR-ENTRAINED									
H........	1½-in. gravel	4.0	28	0.48	0.61	0.81	1.01	1.00	...
NORMAL WEIGHT—NON AIR-ENTRAINED—CURED 7 DAYS MOIST, 21 DAYS DRY—TESTED DRY									
G.........	1-in. gravel	2.5	28	0.32	0.52	0.70	0.93	1.00	1.04
NORMAL WEIGHT—AIR-ENTRAINED									
C........	1½-in. gravel	7.0	28	0.54	0.72	0.84	0.96	1.00	...
	1½-in. gravel	7.0	28	0.55	0.75	0.95	0.99	1.00	...
E........	1-in. gravel	4.5	7	0.61	0.87	0.96	1.03	1.00	...
	1-in. gravel	4.5	91	0.52	0.78	0.94	1.04	1.00	...
I..........	¾-in. stone	6.0	28	0.43	0.64	0.79	0.90	1.00	0.94
	Average..........................			0.53	0.75	0.90	0.98	1.00	0.94
LIGHTWEIGHT									
I..........	½-in. expanded clay	6.0	28	0.60	0.82	0.88	0.93	1.00	0.89

All specimens moist cured until tested unless otherwise noted.

TABLE II.—STRENGTH CORRECTION FACTORS FOR 2000-psi CONCRETE.

Cooperating Laboratories	Aggregate	Slump, in.	Age, days	l/d Ratio					
				0.50	0.75	1.00	1.50	2.00	2.50
Molded Cylinders—6-in. diameter (unless noted otherwise)									
Normal weight—non air-entrained									
A	1-in. stone	2.5	7	0.60	0.65	0.82	0.94	1.00	...
	1-in. stone	2.5	91	0.56	0.64	0.65	0.80	1.00	...
D	1¼-in. stone	3.0	28	0.45	0.64	0.75	0.91	1.00	...
H	1½-in. gravel	4.0	28	0.60	0.79	0.87	1.06	1.00	...
	Average			0.55	0.68	0.72	0.93	1.00	...
Normal weight—3-in. diameter—non air-entrained									
D	1¼-in. stone	3.0	28	0.45	0.61	0.77	0.97	1.00	...
Normal weight—non air-entrained—cured 7 days moist, 21 days dry—tested dry									
G	1-in. gravel	2.5	28	0.44	0.67	0.81	0.94	1.00	1.04
	1-in. gravel	2.5	28	0.41	0.60	0.76	0.91	1.00	1.01
	Average			0.43	0.64	0.79	0.93	1.00	1.03
Normal weight—4-in. diameter—non air-entrained—cured 7 days moist, 21 days dry—tested dry									
G	1-in. gravel	2.5	28	0.43	0.72	0.88	0.92	1.00	...
	1-in. gravel	6.0	28	0.34	0.63	0.87	0.91	1.00	...
	Average			0.39	0.68	0.88	0.92	1.00	...
Normal weight—autoclaved									
C	1½-in. stone	6.0	...	0.48	0.60	0.73	0.92	1.00	...
	1½-in. stone	5.5	...	0.45	0.61	0.75	0.92	1.00	...
	Average			0.47	0.61	0.74	0.92	1.00	...
Normal weight—air-entrained									
B	1-in. slag	2.5	7	0.71	0.86	0.91	1.01	1.00	...
	1-in. slag	2.5	28	0.72	0.84	0.90	1.01	1.00	...
C	1½-in. gravel	6.0	28	0.63	0.80	0.91	0.99	1.00	...
	1½-in. gravel	6.0	28	0.65	0.83	0.90	0.94	1.00	...
E	1-in. gravel	4.5	7	0.71	0.89	0.97	1.04	1.00	...
	1-in. gravel	4.5	91	0.64	0.89	0.93	1.00	1.00	...
F	1½-in. stone	5.0	28	0.56	0.71	0.81	0.94	1.00	...
	1½-in. stone	2.5	28	0.62	0.75	0.86	0.95	1.00	...
I	¾-in. stone	6.0	28	0.54	0.75	0.84	1.02	1.00	1.04
	¾-in. stone	6.0	28	0.64	0.80	0.84	0.91	1.00	0.99
	1½-in. stone	6.0	28	0.70	0.83	0.88	1.02	1.00	1.02
	1½-in. stone	6.0	28	0.70	0.81	0.88	0.98	1.00	1.01
	Average			0.65	0.81	0.89	0.98	1.00	1.02

TABLE II.—*Concluded.*

Cooperating Laboratories	Aggregate	Slump, in.	Age, days	*l/d* Ratio					
				0.50	0.75	1.00	1.50	2.00	2.50
LIGHTWEIGHT—100 TO 116 LB PER CU FT (APPROXIMATELY)									
B	½-in. expanded slag	2.5	7	0.82	0.89	0.95	1.01	1.00	...
	½-in. expanded slag	2.5	28	0.78	0.86	0.93	1.00	1.00	...
D	¾-in. expanded slag	6.0	28	0.74	0.84	0.83	0.98	1.00	...
I	½-in. expanded clay	6.0	28	0.85	0.93	0.92	0.83	1.00	0.98
	Average			0.80	0.88	0.91	0.95	1.00	0.98
CORES—NORMAL WEIGHT									
6-IN. DIAMETER									
A	1-in. stone	2.5	7	0.54	...	0.77	...	1.00	...
	1-in. stone	2.5	28	0.67	...	0.71	...	1.00	...
I	¾-in. stone	6.0	28	0.50	0.84	0.86	1.04	1.00	0.97
	1½-in. stone	6.0	28	0.49	0.63	0.84	1.00	1.00	1.04
	Average			0.53	0.74	0.80	1.02	1.00	1.01
3-IN. DIAMETER									
I	¾-in. stone	6.0	28	0.55	0.71	0.88	1.06	1.00	0.91
	1½-in. stone	6.0	28	0.56	0.85	0.89	0.99	1.00	1.13
	Average			0.56	0.78	0.89	1.03	1.00	1.02

All specimens moist cured until tested unless otherwise noted.

ital letters, A through J. The letters have no significance, but they do enable the reader to locate in the tables all the data from any single laboratory for the purpose of determining the effect of the variables studied by that laboratory.

TEST RESULTS

The more important results are presented in Tables I through V. Since strength is an accepted important variable, all data for concrete of the same nominal strength are included in the same table. Subheadings within the tables are used to designate the variables considered. The major part of the data appears in Tables II, III, and IV, representing nominal concrete strengths of 2000, 3500, and 5000 psi, respectively. A few tests were made on 1000 and 7000 psi concrete; these data are given in Tables I and V, respectively. Each correction factor given is based on at least three individual tests.

Since numerous concrete mixes were used, no attempt has been made to present individual mix data; however, in each case the type of aggregate used and the slump are given. Some laboratories reported the slump for each mix while others reported only the nominal values; however, in most instances the values given are correct within ±½-in.

Except for specimens made by laboratories C, G, and J, all specimens were moist cured until tested and tested wet. Some laboratories molded specimens to the correct height, others sawed them. Different capping materials were used. No attempt has been made to study the

TABLE III.—STRENGTH CORRECTION FACTORS FOR 3500-psi CONCRETE.

Cooperating Laboratories	Aggregate	Slump, in.	Age, days	*l/d* Ratio					
				0.50	0.75	1.00	1.50	2.00	2.50
Molded Cylinders—6-in. diameter unless noted otherwise									
Normal weight—non air-entrained									
A	1-in. stone	2.5	7	0.72	0.78	0.89	0.94	1.00	...
	1-in. stone	2.5	91	0.83	0.86	0.87	0.95	1.00	...
D	1¼-in. stone	4.0	28	0.61	0.75	0.84	0.96	1.00	...
H	1½-in. gravel	4.0	28	0.63	0.78	0.87	0.93	1.00	...
	Average			0.70	0.79	0.87	0.95	1.00	...
Normal weight—3-in. diameter—non air-entrained									
D	1¼-in. stone	4.0	28	0.58	0.72	0.94	1.11	1.00	...
Normal weight—non air-entrained—cured 7 days moist, 21 days dry—tested dry									
G	1-in. gravel	2.5	28	0.43	0.65	0.79	0.92	1.00	0.98
	1-in. gravel	2.5	28	0.53	0.68	0.81	0.86	1.00	1.03
	Average			0.48	0.67	0.80	0.89	1.00	1.01
Normal weight—4-in. diameter—non air-entrained—cured 7 days dry, 21 days wet—tested dry									
G	1-in. gravel	2.5	28	0.45	0.66	0.72	0.89	1.00	...
Normal weight—autoclaved									
C	1½-in. stone	6.0	...	0.59	0.71	0.83	0.97	1.00	...
	1½-in. stone	6.2	...	0.54	0.70	0.82	0.99	1.00	...
	Average			0.57	0.71	0.83	0.98	1.00	...
Normal weight—air-entrained									
B	1-in. slag	2.5	7	0.79	0.87	0.96	0.99	1.00	...
	1-in. slag	2.5	28	0.76	0.86	0.92	1.00	1.00	...
C	1½-in. gravel	5.7	28	0.73	0.84	0.88	0.95	1.00	...
	1½-in. gravel	6.0	28	0.71	0.87	0.91	0.98	1.00	...
E	1-in. gravel	4.2	7	0.69	0.83	0.91	1.03	1.00	...
	1-in. gravel	4.5	7	0.69	0.82	0.89	0.99	1.00	...
	1-in. gravel	4.5	7	0.67	0.81	0.88	1.01	1.00	...
	1-in. gravel	4.4	91	0.66	0.82	0.90	1.00	1.00	...
F	1½-in. stone	5.0	28	0.67	0.79	0.82	0.95	1.00	...
	1½-in. stone	2.5	28	0.66	0.79	0.86	0.97	1.00	...
I	¾-in. stone	6.0	28	0.64	0.81	0.89	1.00	1.00	1.04
	¾-in. stone	6.0	28	0.72	0.82	0.90	0.95	1.00	0.97
	1½-in. stone	6.0	28	0.69	0.84	0.93	0.96	1.00	0.98
	1½-in. stone	6.0	28	0.68	0.78	0.87	0.97	1.00	1.00
	Average			0.70	0.83	0.89	0.98	1.00	1.00

TABLE III.—*Concluded.*

Cooperating Laboratories	Aggregate	Slump, in.	Age, days	*l/d* Ratio					
				0.50	0.75	1.00	1.50	2.00	2.50
LIGHTWEIGHT—100 TO 116 LB PER CU FT (APPROXIMATELY)									
B	½-in. expanded slag	2.5	7	0.82	0.89	0.92	0.99	1.00	...
	½-in. expanded slag	2.5	28	0.80	0.86	0.87	0.99	1.00	...
D	¾-in. expanded slate	3.0	28	0.80	0.82	0.85	0.87	1.00	...
I	½-in. expanded clay	6.0	28	0.84	0.90	0.98	0.92	1.00	1.00
	Average			0.82	0.87	0.91	0.94	1.00	1.00
CORES—NORMAL WEIGHT									
6-IN. DIAMETER									
A	1-in. stone	2.5	7	0.64	...	0.77	...	1.00	...
	1-in. stone	2.5	91	0.69	...	0.90	...	1.00	...
I	¾-in. stone	6.0	28	0.55	0.76	0.83	0.97	1.00	0.96
	1½-in. stone	6.0	28	0.61	0.76	0.88	1.02	1.00	0.93
	Average			0.62	0.76	0.85	1.00	1.00	0.95
3-IN. DIAMETER									
I	¾-in. stone	6.0	28	0.69	0.68	0.84	0.94	1.00	1.08
	1½-in. stone	6.0	28	0.52	0.65	0.77	1.03	1.00	1.06
	Average			0.61	0.67	0.81	0.99	1.00	1.07

All specimens moist cured until tested unless noted otherwise.

influence the latter two variables may have on the correction factors.

Effect of Strength:

The effect of strength on the correction factors is shown in Figs. 2 and 3. Figure 2 is plotted from the data for normal-weight air-entrained concrete. Except for the 1000 and 2000 psi concretes, the non-air-entrained normal-weight concretes have nearly the same correction factors as the air-entrained concretes. For these weaker concretes, significantly more correction is needed for the nonair-entrained concrete. Figure 3 was plotted from the data for the lightweight concretes. The dashed line in each figure represents correction factors from ASTM Methods C 42. These figures clearly indicate that concrete strength is quite important in determining the correction factors for the low *l/d* ratios and that the lower strength concretes need a greater correction than do the higher strength concretes.

Effect of Aggregates:

A comparison of the data in the tables or in Figs. 2 and 3 shows that concrete made with lightweight aggregates need considerably less correction for a given strength for the different *l/d* ratios than does the normal-weight concrete. The lightweight concretes tested were in the 100 to 116 lb per cu ft range.

The data indicate that there is no significant difference between concretes made of naturally occurring gravel and those made with crushed stone; however, the correction factors for normal-weight air-entrained concrete made of air-cooled

TABLE IV.—STRENGTH CORRECTION FACTORS FOR 5000-psi CONCRETE.

Cooperating Laboratories	Aggregate	Slump, in.	Age, days	l/d Ratio					
				0.50	0.75	1.00	1.50	2.00	2.50
MOLDED CYLINDERS—6-IN. DIAMETER (UNLESS NOTED OTHERWISE)									
NORMAL WEIGHT—NON AIR-ENTRAINED									
A	1-in. stone	2.5	7	0.71	0.78	0.85	0.90	1.00	...
	1-in. stone	2.5	91	0.80	0.83	0.85	0.89	1.00	...
D	1¼-in. stone	4.0	28	0.67	0.81	0.90	0.98	1.00	...
H	1½-in. gravel	4.0	28	0.87	0.91	0.93	0.96	1.00	...
	Average			0.76	0.83	0.88	0.93	1.00	...
NORMAL WEIGHT—3-IN. DIAMETER—NON AIR-ENTRAINED									
D	1¼-in. stone	4.0	28	0.56	0.72	0.88	1.10	1.00	...
NORMAL WEIGHT—NON AIR-ENTRAINED—CURED 7 DAYS MOIST, 21 DAYS DRY—TESTED DRY									
G	1-in. gravel	2.5	28	0.55	0.70	0.78	0.90	1.00	1.02
	1-in. gravel	2.5	28	0.54	0.66	0.75	0.92	1.00	0.96
	Average			0.55	0.68	0.77	0.91	1.00	0.99
NORMAL WEIGHT—4-IN. DIAMETER—NON AIR-ENTRAINED—CURED 7 DAYS MOIST, 21 DAYS DRY—TESTED DRY									
G	1-in. gravel	2.5	28	0.58	0.74	0.80	0.90	1.00	...
NORMAL WEIGHT—AUTOCLAVED									
C	1½-in. gravel	4.0	...	0.59	0.75	0.80	0.91	1.00	...
	1½-in. gravel	3.4	...	0.58	0.71	0.80	0.95	1.00	...
	Average			0.59	0.73	0.80	0.93	1.00	...
NORMAL WEIGHT—AIR-ENTRAINED									
B	1-in. slag	2.5	7	0.80	0.92	0.94	1.00	1.00	...
	1-in. slag	2.5	28	0.78	0.87	0.93	1.00	1.00	...
C	1½-in. gravel	2.3	28	0.68	0.82	0.88	0.96	1.00	...
	1½-in. gravel	2.2	28	0.71	0.85	0.87	0.94	1.00	...
E	1-in. gravel	4.3	91	0.65	0.80	0.87	0.95	1.00	...
	1-in. gravel	4.3	91	0.61	0.78	0.86	0.99	1.00	...
	1-in. gravel	4.5	91	0.63	0.78	0.86	0.98	1.00	...
F	1½-in. stone	5.0	28	0.81	0.90	0.93	0.98	1.00	...
	1½-in. stone	2.5	28	0.71	0.80	0.88	0.96	1.00	...
I	¾-in. stone	2.5	28	0.92	0.89	0.94	0.97	1.00	1.10
	¾-in. stone	2.5	28	0.75	0.84	0.82	0.92	1.00	0.95
	1½-in. stone	2.5	28	0.58	0.73	0.96	1.02	1.00	1.01
	1½-in. stone	2.5	28	0.71	0.80	0.89	0.96	1.00	0.95
	Average			0.71	0.82	0.89	0.97	1.00	1.00

TABLE IV.—*Concluded.*

Cooperating Laboratories	Aggregate	Slump, in.	Age, days	l/d Ratio					
				0.50	0.75	1.00	1.50	2.00	2.50
LIGHTWEIGHT—100 TO 116 LB PER CU FT (APPROXIMATELY)									
B	½-in. expanded slag	2.5	7	0.82	0.90	0.97	1.00	1.00	...
	½-in. expanded slag	2.5	28	0.83	0.86	0.88	0.95	1.00	...
D	¾-in. expanded slate	3.5	28	0.95	0.97	0.96	1.00	1.00	...
I	½-in. expanded clay	2.5	28	0.93	0.90	0.93	0.95	1.00	1.03
	Average			0.88	0.91	0.94	0.98	1.00	1.03
CORES—NORMAL WEIGHT									
6-IN. DIAMETER									
A	1-in. stone	2.5	7	0.77	...	0.82	...	1.00	...
	1-in. stone	2.5	91	0.76	...	0.84	...	1.00	...
I	¾-in. stone	2.5	28	0.66	0.73	0.84	0.97	1.00	0.96
	1½-in. stone	2.5	28	0.58	0.73	0.96	1.02	1.00	1.01
J	1½-in. stone	2.5	733	...	0.63	0.77	0.94	1.00	...
	Average			0.69	0.70	0.85	0.98	1.00	0.99
3-IN. DIAMETER									
I	¾-in. stone	2.5	28	0.63	0.68	0.86	1.08	1.00	1.10
	1½-in. stone	2.5	28	0.55	0.77	0.77	1.01	1.00	1.15
	Average			0.59	0.73	0.82	1.05	1.00	1.13

All specimens moist cured until tested unless otherwise noted.

slag appear to be slightly higher than those for the gravel or crushed stone. The weight of the slag concrete was approximately 136 lb per cu ft.

The increase in the magnitude of the correction factors as the weight of the concrete decreases suggests the possibility of correlating the correction factors with density as well as with strength. Sufficient evidence is not now available to determine if this would be a profitable approach.

Laboratory I made specimens using both a ¾-in. and 1½-in. crushed stone. There are no significant differences in the results obtained.

Effect of Diameter:

The general behavior of specimens having 3- or 4-in. diameter was the same as for specimens having the standard 6-in. diameter. However, the smaller diameter molded specimens require somewhat greater corrections at the lower l/d values than do the standard diameter specimens. The correction factors for cores seem to be about the same whether the diameter is 3 in. or 6 in. Comparisons may be made by studying the data of laboratories D, J, and I.

Effect of Age and Curing:

For specimens continually moist cured there is little difference in the correction factors regardless of age. In some cases there appears to be a trend which shows that the oldest concretes need the great-

TABLE V.—STRENGTH CORRECTION FACTORS FOR 7000-psi CONCRETE.

Cooperating Laboratory	Aggregate	Slump, in.	Age, days	*l/d* Ratio					
				0.50	0.75	1.00	1.50	2.00	2.50
MOLDED CYLINDERS—6-IN. DIAMETER (UNLESS NOTED OTHERWISE).									
NORMAL WEIGHT—NON AIR-ENTRAINED									
H.........	1½-in. gravel	4.0	28	0.87	0.90	0.92	0.96	1.00	...
NORMAL WEIGHT—AUTOCLAVED									
C.........	1½-in. gravel	3.5	...	0.69	0.80	0.84	0.93	1.00	...
	1½-in. gravel	3.0	...	0.69	0.79	0.83	0.98	1.00	...
	Average..........................			0.69	0.80	0.84	0.96	1.00	...
NORMAL WEIGHT—AIR-ENTRAINED									
C.........	1½-in. gravel	2.1	28	0.78	0.88	1.00	1.10	1.00	...
	1½-in. gravel	2.1	28	0.80	0.93	0.94	1.03	1.00	...
I.........	¾-in. stone	2.5	28	0.78	0.84	0.88	0.87	1.00	1.02
	Average..........................			0.79	0.88	0.94	1.00	1.00	1.02
LIGHTWEIGHT									
I.........	½-in. expanded clay	2.5	28	0.97	0.95	0.99	0.97	1.00	0.96

All specimens moist cured until tested unless otherwise noted.

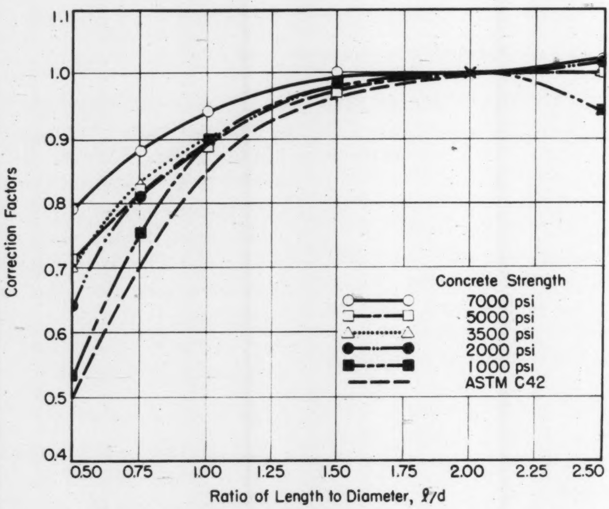


FIG. 2.—Correction Factors for Normal Weight, Air-Entrained Concretes—6-in. Diameter Specimens.

est correction. The data from laboratories A, B, E, and I indicate this trend.

Laboratory C autoclaved some specimens in which the cementing compounds were portland cement, 60 per cent, and silica flour, 40 per cent. The autoclaved specimens were tested when they had reached room temperature, shortly after their removal from the autoclave. Laboratory G cured its specimens 7 days moist and then 21 days in the normal laboratory atmosphere and tested them dry. In both cases greater corrections for all *l*/*d* ratios are needed than for moist-cured specimens tested wet. If it may be assumed that the dry or semi-dry condition of the specimen at the time of testing was more important than the curing,

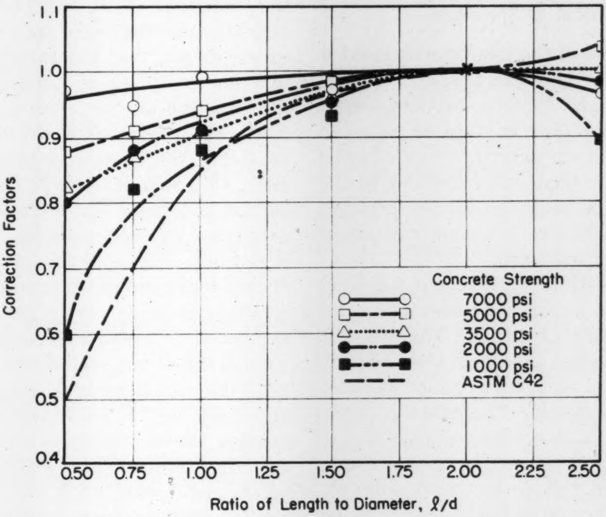


FIG. 3.—Correction Factors for Lightweight Concretes—6 in. Diameter Specimens.

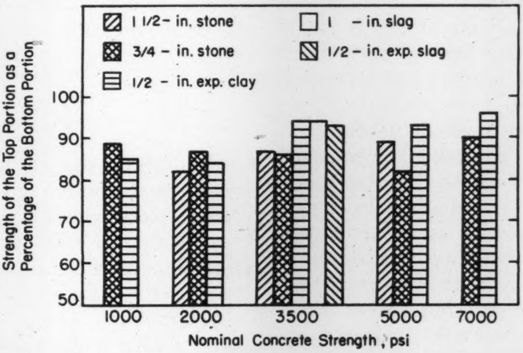


FIG. 4.—Comparison of the Strength of the Top Portion of the Specimen to the Strength of the Bottom Portion—Data from Laboratories B and I.

then it is clear that all specimens should be tested in the same moisture condition when comparisons are to be made.

Effect of Position of Specimen:

While some of the laboratories molded specimens to the correct length, laboratories B, C, and I sawed standard length specimens to obtain the desired *l/d* ratio. When sawed specimens are used, the question is immediately raised as to the effect of obtaining the short specimen from either the top or bottom portion of the standard specimen.

In Tables II to V there are two lines of correction factors for laboratory C for each condition of curing. The top line represents values obtained when the top portion of the standard cylinder was used to obtain specimens of *l/d* ratios less than 2; the bottom line, when the lower portion was used. There is no consistent difference in the correction values obtained for the top or bottom portions for either the 28 days moist-cured or the autoclaved concrete.

The results from laboratories B and I, Fig. 4, indicate that the specimens from the top portion are 4 to 18 per cent weaker than those from the bottom portion. If this were consistently true then the choice of the portion of the standard cylinder from which the shorter specimens were sawed would affect the magnitude of the correction factors obtained. The mixes and handling procedures in laboratory C may have been such that little or no water gain resulted, in which case no difference would be noted between the top and bottom portions.

Effect of Slump:

While there was some variation in slump, only in the results of one laboratory, F, are direct comparisons possible. These meager results do not show that slump affects the correction factors.

Effect of Coring:

Laboratories A, I, and J tested cores made from blocks of concrete cast from the same mix as all or a portion of the molded cylinders tested by those laboratories, except that laboratory J did not test molded cylinders. Laboratory J cast the concrete on the ground the last of April, 1957, and drilled the cores in November, 1958. They were tested May 12, 1959. All cores were made from normal-weight concretes. Laboratories I and J used air-entrained concrete; laboratory A did not. The results from laboratories A and J indicate that the core specimens require greater correction than do molded cylinders. The data from laboratory I, however, shows very definitely that the core specimens require greater correction for the low *l/d* ratios. The discussion above applies to 6-in. diameter cylinders. Laboratory I tested some 3-in. cores and found that the corrections needed were, in general, about the same as for the 6-in. cores. The 3-in. cores yielded slightly higher strengths than the 6-in. cores but less than the 6-in. cylinders, except for the 2000-psi concrete.

SUMMARY

Although the data in this investigation was treated simply, the following conclusions seem evident:

1. As the *l/d* ratio decreases, greater corrections must be made to approximate the strength of the standard cylinders.
2. The lower strength concretes require greater corrections than do the higher strength concretes.
3. Air - entrained concretes require slightly less correction than do the non-air-entrained concretes.
4. Considerably less correction is needed for the lightweight concretes than for the normal-weight concretes.
5. No consistent difference was noted

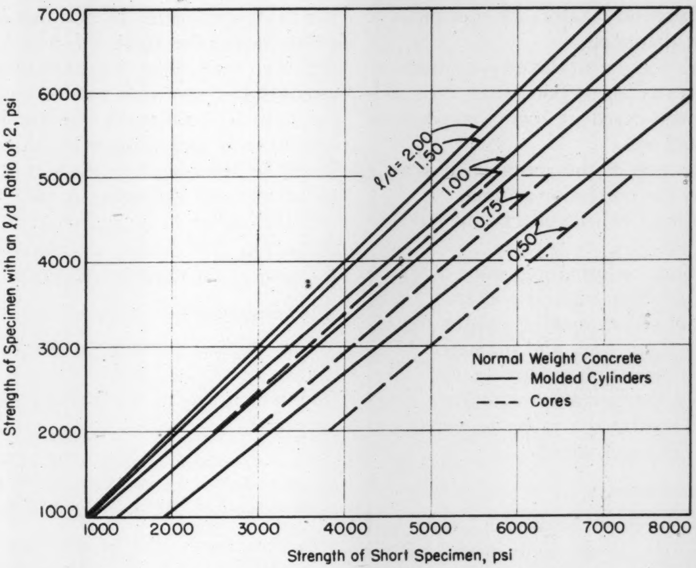


FIG. 5.—Graph for Correcting the Strength of Short Specimens to That of a Specimen with an *l/d* Ratio of 2—Normal Weight Concrete.

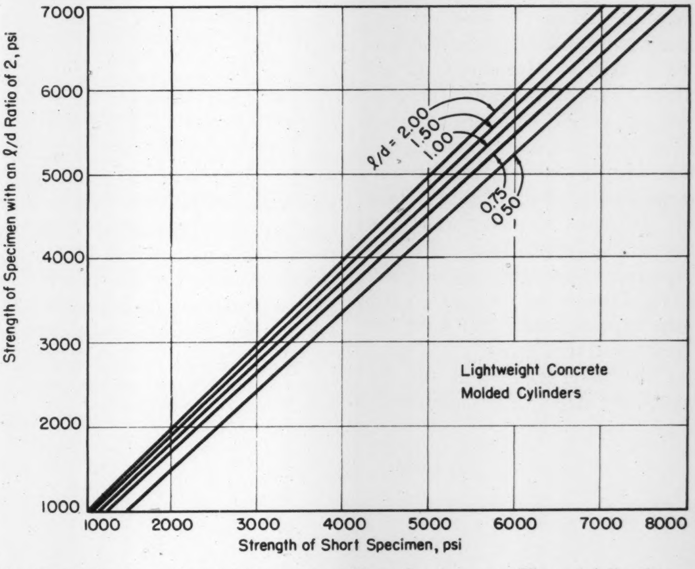


FIG. 6.—Graph for Correcting the Strength of Short Specimens to That of a Specimen with an *l/d* Ratio of 2—Lightweight Concrete.

in the correction factors for specimens of different diameters.

6. Dried and autoclaved specimens require greater corrections than do specimens moist-cured at room temperature and tested wet.

7. The age of the specimen has little effect on the correction factors.

8. Slump has little effect on correction factors.

Additional information, some of which may be obtained from data already available, may aid in making clearer the effects of slump, age, and diameter. Considerable variation occurs in the data, especially for specimens with low *l/d* ratios, and no absolute values of corrections factors can be obtained.

Recommendations:

The correction factors obtained will probably not be generally useful for research purposes; however, they are convenient for obtaining an indication of the standard cylinder strength from short specimens obtained in the field or elsewhere. Since the table of correction factors given in ASTM Methods C 42 are not satisfactory for all concrete strengths or for different weight concretes, Figs. 5 and 6, or similar ones, are recommended for normal weight and lightweight concrete, respectively. Both figures show data for specimens moist-cured and tested wet. The solid lines refer to molded specimens and the dashed lines to cores. The corrections for an *l/d* ratio of 1.50 are approximately the same for molded and cored specimens.

To use the diagrams, enter on the abscissa with the strength obtained from the short specimens. Directly above this point locate the correct *l/d* ratio. The ordinate will then be the estimated strength of a specimen having an *l/d* ratio of 2. If the diameter of the original specimens is something other than 6 in., it will be necessary to use another factor to correct the strength to that of the standard 6 by 12-in. cylinder. For this correction the reader is referred to a previous study reported by Price.[5]

Acknowledgment:

The following laboratories and individuals cooperated in providing the data from which this brief summary has been prepared:

Duquesne Slag Products Co.—H. T. Williams,
Lehigh Portland Cement Co.—S. B. Helms,
Mount Royal Paving and Supplies, Ltd.—N. G. Zoldners,
National Ready Mix Concrete Assn.—D. L. Bloem,
North Carolina State Highway Commission—C. E. Proudly,
Pennsylvania Department of Highways—L. N. Heagy,
Pittsburgh Testing Laboratory—H. F. Hedderich,
Portland Cement Assn.—Paul Klieger,
U. S. Army Engineers Waterways Experiment Station—T. B. Kennedy, and
University of Illinois—C. E. Kesler.

The information and suggestions given the author were quite important and greatly appreciated. The author is particularly indebted to Mr. Helms, who is also chairman of Subcommittee III-a, for his assistance throughout the project and in the preparation of this paper.

---

[5] Walter H. Price, "Factor Influencing Concrete Strength," *Proceedings*, Am. Concrete Inst., Vol. 47, p. 426 (1951).

DISCUSSION

Mr. I. A. Benjamin.[1]—I would like to ask the author what types of concrete he is talking about when he refers to lightweight concrete.

Mr. C. E. Kesler (*author*).—These concretes have weights of approximately 100 to 116 lb per cu ft.

Mr. Benjamin.—What type of aggregates?

Mr. Kesler.—I cannot give you the trade names of these aggregates because I do not know what they all were. We had an expanded slag, an expanded clay and an expanded slate in the lightweight concretes.

Mr. Charles Vollick.[2]—I would like to ask the author if he has any correlation between the 6 by 6 cube strengths or other sizes of cubes and the data he has accumulated on cylinder strengths.

Mr. Kesler.—I do not have any data with me, there is some data available in the literature. I have not tested any 6 by 6-in. cubes but you will find in one of the references to this paper some data on correlation factors between strengths of different specimen types compiled by RILEM. Some factors are given for comparing our standard cylinder to the normal 6 by 6-in. cube or 20 by 20-cm cube.

---

[1] Director of Research, Granco Steel Products, St. Louis, Mo.

[2] Chief, Engineering Laboratories, SIKA Chemical Corp., Passaic, N. J.

STRESSES IN DEEP BEAMS SUBJECTED TO CENTRAL AND THIRD-POINT LOADING*

BY M. E. RAVILLE[1] AND F. J. MCCORMICK[1]

SYNOPSIS

Many workers have noted that values of the modulus of rupture for concrete when computed from tests on beams subjected to a central load are consistently different from values based on beams subjected to third-point loading. Attempts have been made to explain this difference. In the present paper it is shown that for beams so loaded but subjected to the same maximum bending moment, the maximum longitudinal stresses at the lower surface differ significantly. All results are based on the elastic theory, as is the modulus of rupture formula.

In order to obtain a comparison of the stresses in a rectangular beam of the proportions used in standard modulus of rupture tests for concrete, the problem of the rectangular beam was solved by the methods of the theory of elasticity for the two cases of central and third-point loading. Expressions for normal and shearing stresses were determined from a trigonometric series solution. The method used yields relatively simple results, although it does not exactly satisfy the conditions of simple supports at the ends. As the modulus of rupture is computed for the section of maximum bending moment, the exact distribution of stress at the ends is unimportant, according to the principle of St. Venant. The series method employed yields reliable solutions at the significant sections and solutions that are amenable to machine computation. Computations were made for 11 points throughout the depth of the beam at 5 different sections. Since fracture occurs at or near the center section, only values at this section are presented.

MATHEMATICAL ANALYSIS

Figure 1 shows the dimensions and loading for the two cases considered. The assumption that the beams are supported by forces distributed over the ends is made in order to simplify the analysis. Later, it will be shown that the manner in which the ends of the beams are supported has practically no effect on the stress distribution in the middle third of beams having the depth-length ratios considered in this paper. The solutions are based on the equations of two-dimensional elasticity in which stress functions in the form of Fourier series are used. The procedure to be followed in such analyses is well established (**1**).[2] This procedure will be outlined for the case of central loading; only a slight modification is needed for the case of third-point loading.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Professor of Applied Mechanics, Kansas State University, Manhattan, Kans.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The problem is to determine a stress function that satisfies the biharmonic differential equation and at the same time yields stresses that satisfy the conditions at the boundaries of the beam. The stress function is taken in the form

$$\phi = \sum_{m=1,3,5\cdots}^{\infty} f_m(y) \cos \alpha_m x \quad ;$$

$$\text{where: } \alpha_m = \frac{m\pi}{l}.$$

The function $f_m(y)$ is determined by requiring that the stress function ϕ satisfy the equation

$$\nabla^4 \phi = 0$$

The resulting expression for $f_m(y)$ consists of four terms containing hyperbolic functions of y and four integration constants. The stresses in the beam can now be expressed in terms of these four integration constants from the relations:

$$\sigma_x = \frac{\partial^2 \phi}{\partial y^2}; \qquad \sigma_y = \frac{\partial^2 \phi}{\partial x^2}, \quad \text{and} \quad \tau_{xy} = -\frac{\partial^2 \phi}{\partial x\, \partial y}$$

The four constants in these stress equations are evaluated by requiring that the boundary conditions at the top and bottom of the beam be satisfied, that is,

$$\tau_{xy}\Big|_{y=\pm c} = 0; \qquad \sigma_y\Big|_{y=c} = 0;$$

$$\sigma_y \Bigg|_{y=-c} = -\frac{2P}{l} \sum_{m=1,3,5,\cdots}^{\infty} \cos \alpha_m x$$

The series representing $\sigma_y \big|_{y=-c}$ sums to zero for all values of x in the range $-\frac{l}{2} \leq x \leq +\frac{l}{2}$ except $x = 0$; at $x = 0$ the series sums to infinity. The values of the four integration constants having been determined, the expressions for the stresses are now written as follows:

$$\sigma_x = \frac{2P}{l} \sum_{m=1,3,5,\cdots}^{\infty} \Phi_m \cos \alpha_m x \ldots\ldots (1)$$

$$\sigma_y = -\frac{2P}{l} \sum_{m=1,3,5,\cdots}^{\infty} \Psi_m \cos \alpha_m x \ldots.(2)$$

$$\tau_{xy} = \frac{2P}{l} \sum_{m=1,3,5,\cdots}^{\infty} \Delta_m \sin \alpha_m x \ldots\ldots (3)$$

$$\Phi_m = \frac{(\alpha_m c \cosh \alpha_m c - \sinh \alpha_m c) \cosh \alpha_m y - \alpha_m y \sinh \alpha_m c \sinh \alpha_m y}{\sinh 2\alpha_m c + 2\alpha_m c} - \frac{(\alpha_m c \sinh \alpha_m c - \cosh \alpha_m c) \sinh \alpha_m y - \cosh \alpha_m c \cosh \alpha_m y}{\sinh 2\alpha_m c - 2\alpha_m c}$$

$$\Psi_m = \frac{(\alpha_m c \cosh \alpha_m c + \sinh \alpha_m c) \cosh \alpha_m y - \alpha_m y \sinh \alpha_m c \sinh \alpha_m y}{\sinh 2\alpha_m c + 2\alpha_m c} - \frac{(\alpha_m c \sinh \alpha_m c + \cosh \alpha_m c) \sinh \alpha_m y - \alpha_m y \cosh \alpha_m c \cosh \alpha_m y}{\sinh 2\alpha_m c - 2\alpha_m c}$$

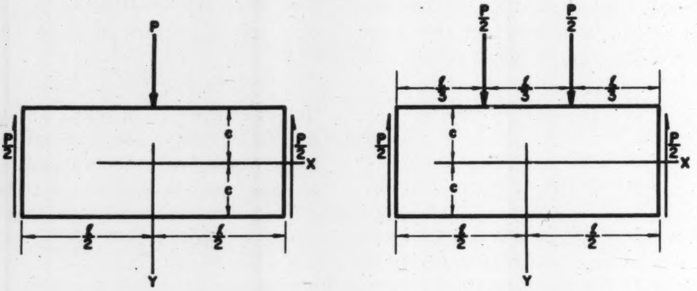
$$\Delta_m = \frac{\alpha_m c \cosh \alpha_m c \sinh \alpha_m y - \alpha_m y \sinh \alpha_m c \cosh \alpha_m y}{\sinh 2\alpha_m c - 2\alpha_m c} - \frac{\alpha_m c \sinh \alpha_m c \cosh \alpha_m y - \alpha_m y \cosh \alpha_m c \sinh \alpha_m y}{\sinh 2\alpha_m c - 2\alpha_m c}$$

Equations 1, 2, and 3 satisfy not only the boundary conditions at the top and bottom of the beam but also the following conditions at the ends of the beam:

$$\sigma_x \Big|_{x=\pm(l/2)} = 0; \qquad \int_{-c}^{c} \tau_{xy} \Bigg|_{x=-(l/2)} dy = \frac{P}{2};$$

$$\int_{-c}^{c} \tau_{xy} \Bigg|_{x=+(l/2)} dy = -\frac{P}{2}$$

Since the governing differential equation and all of the boundary conditions have been satisfied, Eqs 1, 2, and 3 represent the exact mathematical solution of the problem illustrated in Fig. 1(*a*).



(*a*) Central loading. (*b*) Third-point loading.

FIG. 1.—Beams.

The corresponding equations for the beam under third-point loading are obtained by substituting for the boundary condition on σ_y at the top of the beam the expression

$$\sigma_y \Big|_{y=-c} = -\frac{2P}{l} \sum_{m=1,3,5\cdots}^{\infty} \cos\frac{m\pi}{6} \cos \alpha_m x$$

for the expression used in the central loading problem. The resulting equations are:

$$\sigma_x = \frac{2P}{l} \sum_{m=1,3,5\cdots}^{\infty} \Phi_m \cos\frac{m\pi}{6} \cos \alpha_m x \ldots. \quad (4)$$

$$\sigma_y = -\frac{2P}{l} \sum_{m=1,3,5\cdots}^{\infty} \Psi_m \cos\frac{m\pi}{6} \cos \alpha_m x \,.\,. \quad (5)$$

$$\tau_{xy} = \frac{2P}{l} \sum_{m=1,3,5\cdots}^{\infty} \Delta_m \cos\frac{m\pi}{6} \sin \alpha_m x \ldots. \quad (6)$$

where:
Φ_m, Ψ_m, and Δ_m are defined as previously. Equations 4, 5, and 6 represent the exact mathematical solution of the problem illustrated in Fig. 1(*b*).

A large scale digital computer was used in obtaining numerical solutions of the equations for the stresses, Eqs 1 to 6. Values of these stresses were obtained for beams of three different depth-length ratios, namely, $\frac{1}{2}$, $\frac{1}{3}$ and $\frac{1}{4}$. In each case values of the stresses were determined at 11 points on each of 5 different cross-sections, as follows

$$\frac{x}{l} = 0, 0.05, 0.10, \frac{1}{6}, 0.20$$

$$\frac{y}{c} = 0, \pm 0.2, \pm 0.4, \pm 0.6, \pm 0.8, \pm 1$$

Because of symmetry, it was not necessary to consider points on both sides of the center line of the span. The numerical values obtained are believed to be accurate to at least six significant figures, since the series involved converge rather rapidly and all terms large enough to contribute to the eighth significant figure were considered. Certain values of the stresses in the neighborhood of points of application of concentrated loads are open to question since at these points convergence becomes very slow and the values themselves are approaching infinity. At such points overflow in the machine was experienced, as might be expected, and the values in the machine up to the point of overflow were used.

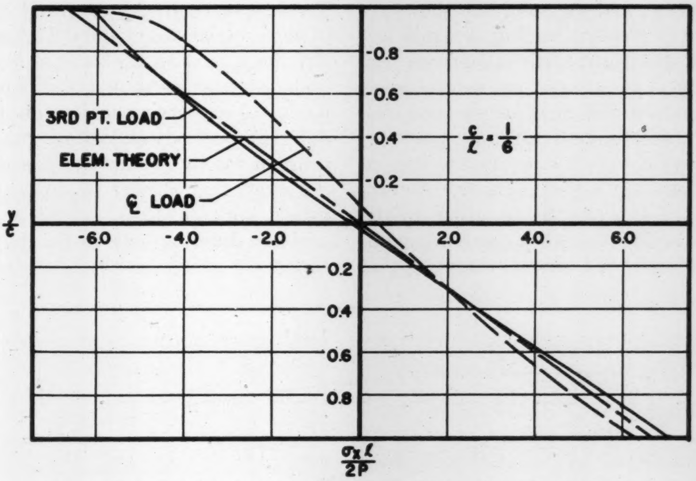


FIG. 2.—Distribution of Longitudinal Normal Stress Over Center Section.

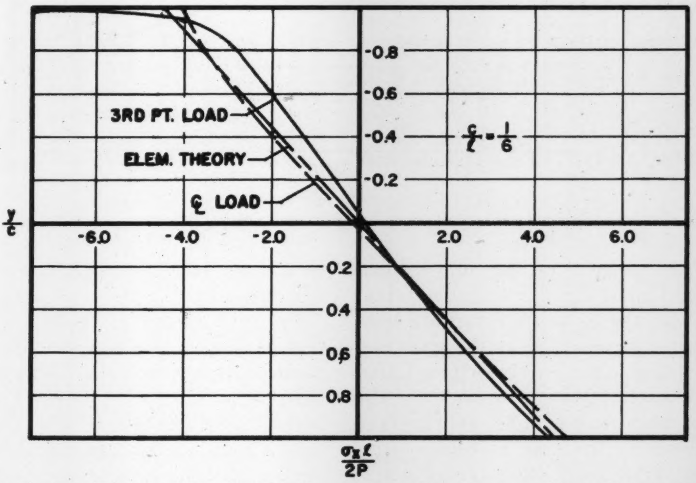


FIG. 3.—Distribution of Longitudinal Normal Stress Over Cross-section at Third Point.

DISCUSSION OF RESULTS

In Fig. 2, the distribution of the bending stress σ_x over the depth at the central cross-section is shown for three cases, each with a depth-length ratio of one third, and each subjected to the same bending moment. The straight line shows the linear distribution predicted by elementary beam theory. The other two lines show the distribution as predicted by the theory of elasticity. Results for center-point loading are shown by the dashed line, and the solid line shows results for third-point loading. A significant shift in the neutral axis is evident, particularly in the case of the central loading. Lindner and Sprague (2) have discussed this at some length.

The value of the maximum normal stress is usually taken as the criterion for failure in concrete as in other brittle materials. This being the case, the significant stress is the normal stress, σ_x, at the lower surface of the mid-section. Examination of Fig. 2 reveals that, compared with the modulus of rupture computation as represented by the lower end of the straight line, the elasticity solution yields a stress 5.94 per cent higher in the case of third-point loading, and 5.50 per cent lower in the case of center point loading; or a total difference of about 11.5 per cent exists between the two cases.

This analysis predicts that, other things being equal and the material remaining elastic to failure, the third-point loading tests will yield modulus of rupture values about 11.5 per cent lower than those resulting from central loading. Tests reported by Lindner and Sprague (2) show variations of about 12.5 per cent indicating that on the basis of their limited test data most of the difference in moduli of rupture resulting from the two different methods of loading can be accounted for by elastic analysis alone.

Pickett and Sherrer (3), in an unpublished work in 1953, solved a similar problem differing only in the method of support in which the results at the center section at the bottom agree with those reported here.

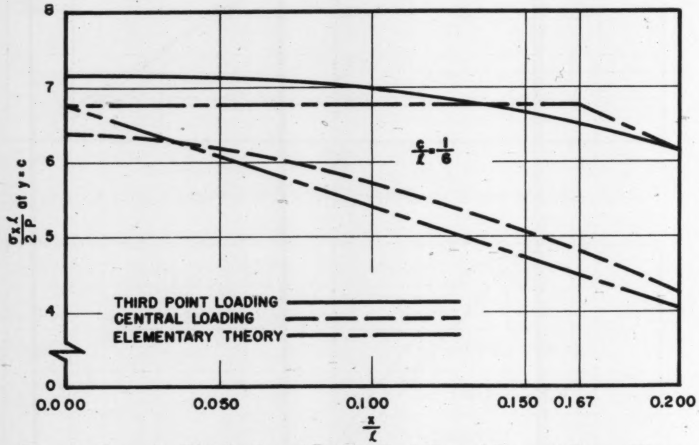


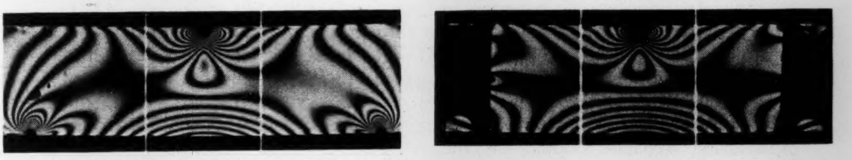
FIG. 4.—Spanwise Distribution of Longitudinal Normal Stress at Lower Fiber.

Figure 3 is similar to Fig. 2 except that in Fig. 3 the cross-section at the third point is considered. Here the elasticity solution yields stresses slightly lower than those given by the elementary theory in the case of third-point loading, and somewhat higher in the case of central loading. The over-all difference in stresses between the central and third-point loadings at this cross-section is less than at the center section and is probably of no particular significance in a discussion of modulus of rupture tests.

The distribution of horizontal normal stress σ_x at the lower boundary from center to third-point is plotted in Fig. 4. For third-point loading, the elementary theory shows the bending stress at the lower surface to be uniform in this region, since the bending moment is constant. The elasticity theory, as illustrated by the solid line, shows a gradually diminishing stress from center to end, but with a fairly constant value in the middle third. For center point loading the elementary theory yields stresses that are maximum at the center and that fall off linearly towards the ends in porportion to the bending moment. Corresponding values given by the theory of elasticity change more slowly in the middle third, and more rapidly in the outer third. The change in σ_x between the center section and third-point section is greater with central than with third-point loading, thus indicating that the third-point loading is more likely to find a weakened section.

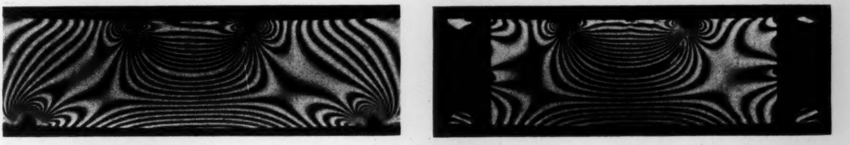
Some photoelastic studies were made in an effort to verify the statement that the distribution of end shear has no significant effect on the maximum bending stress or, in fact, on the stress distribution in the middle third of the beam.

In Fig. 5 are shown two pictures of the same beam supported in two different ways, but with the same total load. In Fig. 5(*a*) the beam is shown resting on rollers, while in Fig. 5(*b*) the ends of the beam were drilled at mid-depth and supported on pins at these points; the vertical lines indicate the third-point sections. Despite the fact that the conditions at the supports are so different, the isochromatic patterns in the middle third of the beams are strikingly similar.



(*a*) Beam resting on rollers. (*b*) Beam supported on pins fitted in drilled ends.

FIG. 5.—Isochromatic Pattern for Central Loading.



(*a*) Beam resting on rollers. (*b*) Beam supported on pins fitted in drilled ends.

FIG. 6.—Isochromatic Pattern for Third-point Loading.

A similar effect is shown in Fig. 6 for the case of third-point loading.

The total load imposed on each of the beams of Fig. 5 was two thirds of that imposed on the beams of Fig. 6 in order to produce the same maximum bending moment at the center section in all four cases.

A shear distribution on the end of the beam such as results from the elasticity analysis presented above would be almost impossible to reproduce, but it is believed that any change in the maximum value of σ_x at the center section produced by such a reaction would be no more significant than that shown in Figs. 5 and 6.

It is concluded that the mathematical

analysis employed in this paper is quite accurate when used to evaluate the significant stress in beams of the depth-length ratio considered.

CONCLUSIONS

It is concluded from the above analysis that, for rectangular beams with a depth-span ratio of one third, center and third-point loading systems cannot be interchanged if a direct comparison of modulus of rupture values is to be made. The solution, based on the theory of elasticity, predicts modulus of rupture values about 11.5 per cent higher for center point loading than for third-point loading. However, some further experimental results should be obtained before this factor is adopted for conversion from one system of loading to the other.

Acknowledgment:

The authors wish to thank S. T. Parker, Director of the IBM 650 Installation, for programming the series solutions and obtaining the numerical results.

REFERENCES

(1) S. Timoshenko and J. N. Goodier, "Theory of Elasticity," McGraw-Hill Book Co., Inc., New York, N. Y., 2nd Edition, p. 46 (1951).

(2) C. P. Lindner and J. C. Sprague, "Effect of Depth of Beam Upon the Modulus of Rupture of Plain Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 1062 (1955).

(3) G. Pickett and R. E. Sherrer, "Simply Supported Beams under Concentrated Loads." Dept. of Mechanics, Univ. of Wisconsin (1953) (*Unpublished*).

DEVELOPMENT OF ASTM STANDARD REFERENCE BLOCKS FOR ULTRASONIC INSPECTION*

BY F. C. PANIAN[1] AND H. E. VANVALKENBURG[2]

SYNOPSIS

A review is given of the investigation which led to the development of the ASTM aluminum alloy ultrasonic standard reference blocks described in the Recommended Practice for Fabricating and Checking Aluminum Alloy Ultrasonic Standard Reference Blocks (E 127 - 58 T).[3] The physical variables affecting the ultrasonic response from the hole or artificial discontinuity in the blocks are discussed. Also included are important considerations that illustrate the need for a fundamental comparison standard and for precise control of parameters and techniques of the ultrasonic tests used to evaluate the stock and the finished reference block.

Most methods for testing materials require equipment calibration through the use of techniques and devices to establish procedures and acceptance standards. Standardization is particularly important in the field of nondestructive testing where conclusions are based upon relative test results which are influenced substantially by the degree of control applied to the test parameters. For example, specifications for radiographic inspection, almost without exception, require the use of penetrameters to insure proper contrast and definition because image quality can vary greatly with the type of equipment, film, and technique employed. Ultrasonic inspection methods also involve a number of parameters which must be controlled by appropriate calibration procedures.

Throughout the metals industry today, ultrasonic flaw detection by the pulse-reflection method is in widespread use to control the internal quality of the product. The pulse-reflection method provides an indication (in the form of a voltage pulse on the screen of a cathode-ray tube) when a discontinuity is detected. The magnitude of the indication roughly represents the amount of pulsed ultrasonic energy that is reflected from the discontinuity and is usually the basis for acceptance or rejection of an inspected part.

The need for suitable calibration methods was recognized almost simultaneously with the introduction of commercial ultrasonic flaw detectors in 1944. Since this equipment employs electronic pulsers and amplifiers with a number of tuning and gain controls as well as selectable probes, or search units, a wide range of test conditions could be chosen by the operator. Sensitivity could be set at one extreme so that minute and possibly insignificant discontinuities were easily detected or, at the other extreme, so that gross discontinuities were barely indicated. Although the early ultrasonic tests were primarily qualitative and required reliable calibration methods only to insure reproducibility, a trend toward using ultrasonic testing as a more quantitative inspection soon developed. The latter inspection requires specified techniques and establishes the final acceptance or rejection of a part as determined by a specified height of indication from a detected discontinuity. In order to maintain agreement between producers and users of metallic products regarding definition of quality, the standardization of these ultrasonic tests to a reliable reference standard becomes mandatory.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Research Engineer, Physical Metallurgy Division, Alcoa Research Laboratories, Aluminum Co. of America, New Kensington, Pa.

[2] Sperry Products, Inc., Danbury, Conn.

[3] 1958 Book of ASTM Standards, Part 3.

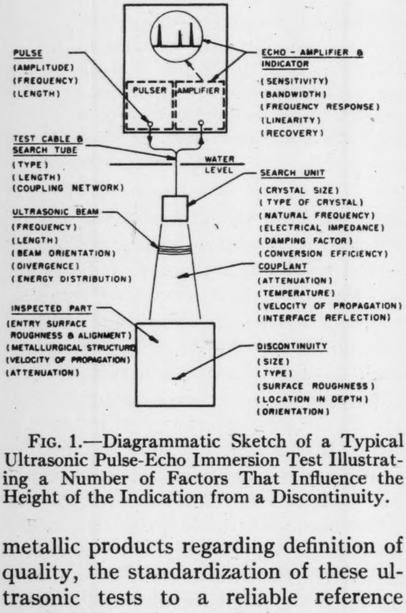

PULSE
(AMPLITUDE)
(FREQUENCY)
(LENGTH)
PULSER
AMPLIFIER
ECHO - AMPLIFIER & INDICATOR
(SENSITIVITY)
(BANDWIDTH)
(FREQUENCY RESPONSE)
(LINEARITY)
(RECOVERY)
TEST CABLE & SEARCH TUBE
(TYPE)
(LENGTH)
(COUPLING NETWORK)
WATER LEVEL
SEARCH UNIT
(CRYSTAL SIZE)
(TYPE OF CRYSTAL)
(NATURAL FREQUENCY)
(ELECTRICAL IMPEDANCE)
(DAMPING FACTOR)
(CONVERSION EFFICIENCY)
ULTRASONIC BEAM
(FREQUENCY)
(LENGTH)
(BEAM ORIENTATION)
(DIVERGENCE)
(ENERGY DISTRIBUTION)
COUPLANT
(ATTENUATION)
(TEMPERATURE)
(VELOCITY OF PROPAGATION)
(INTERFACE REFLECTION)
INSPECTED PART
(ENTRY SURFACE ROUGHNESS & ALIGNMENT)
(METALLURGICAL STRUCTURE)
(VELOCITY OF PROPAGATION)
(ATTENUATION)
DISCONTINUITY
(SIZE)
(TYPE)
(SURFACE ROUGHNESS)
(LOCATION IN DEPTH)
(ORIENTATION)

FIG. 1.—Diagrammatic Sketch of a Typical Ultrasonic Pulse-Echo Immersion Test Illustrating a Number of Factors That Influence the Height of the Indication from a Discontinuity.

The development of practical reference standards for ultrasonic inspection was undertaken in 1951 by Subcommittee VI on Ultrasonic Testing Procedure of ASTM Committee E-7 on Nondestructive Testing. This paper describes the results of a recent program which form the basis for the Tentative Recommended Practice for Fabricating and Checking Aluminum Alloy Ultrasonic Standard Reference Blocks (E 127 – 58 T).[3] Although the project was under the jurisdiction of Subcommittee VI, this paper is not intended primarily as a committee report; an evaluation of both the basic problems involved and the merits of the approach taken by the task group is also included.

VARIABLES LIMITING UNIFORM TEST RESULTS

In order to define clearly the problem of standardizing ultrasonic tests, it is desirable to describe the factors that constitute limitations upon reproducibility. An analysis of a typical pulse-echo immersion test as shown in Fig. 1 will reveal a significant number of the more important variables that influence the height of the indication from a detected discontinuity. Actually, tests by the contact method are also affected by most of the factors that affect immersion tests.

The performance characteristics of both the test instrument and the search unit generally establish the nature of the ultrasonic beam impinging upon the entry surface of the test piece. Referring to Fig. 1, as the pulsed ultrasonic energy is propagated toward the discontinuity the beam configuration and the intensity level are modified by a number of variables in both the couplant and the part. The amount of reflection from the discontinuity depends not only upon the actual intensity of the beam at the discontinuity but also upon the acoustic reflectivity of the surface of the discontinuity. During the return trip, the reflected ultrasound is also affected by the same factors that influenced the transmitted beam.

By employing a suitable standardization technique, the effect of a number of the variables listed in Fig. 1 can be minimized. However, it should be noted that although these variables may be standardized to some extent, they still exist and can impose a serious limitation upon the quantitative value of the test.

Techniques for Test Standardization

While a comprehensive analysis of all aspects of the standardization problem is beyond the scope of this paper, it is pertinent to review briefly the merits and limitations of the following techniques which have been suggested:

1. Provide scales on instrument panel controls so that settings can be duplicated.
2. Employ electrical calibration checks to establish pulser output and receiver amplifier gain.
3. Produce an artificial flaw indication by introducing a target into the beam either between the search unit and specimen or at the rear surface of the specimen.
4. Use the reflection from a geometrical discontinuity normally in the specimen, for example, back surface, keyway, or bore hole.
5. Fabricate a "standard" discontinuity into a noncritical portion of the specimen which later can be machined away or filled by welding.
6. Use one or more separate reference blocks containing simulated discontinuities to establish test sensitivity and equipment performance.

To be widely applicable, the optimum technique should calibrate the entire system including instrument, interconnecting cables, couplant, search unit, positioner, and indicator, recorder, or alarm. Therefore, methods based on electrical measurements only, such as methods 1 and 2, are not satisfactory although they may be very useful for equipment maintenance, repetitive testing, and laboratory studies. The remaining approaches do provide, with some limitations, a system or operational check. Although it would appear feasible in immersed inspection, no practical application of method 3, which involves the use of an artificial target external to the test specimen, has been employed commercially.

Use of the test specimen itself for developing a reference indication as suggested by methods 4 and 5 has certain attractive features, but several major disadvantages may be realized. Frequently there are no satisfactory natural reflecting surfaces which provide a usable range of signal amplitudes, particularly in irregularly shaped objects. Addition of holes, notches, and other similar artificial flaws is generally not permitted in the finished product. Moreover, a fundamental limitation exists whenever significant differences in ultrasonic penetration may occur from specimen to specimen. The standardization is then accurate only at the depth of the artificial flaw unless rather elaborate compensation procedures are employed. However, variations of these methods do meet the requirements for certain inspection applications and are often called for in specifications for ultrasonic inspection of steel tubing, pipe, sheet, and rotor forgings.

Standardization by the use of metallic reference or test blocks containing synthesized discontinuities of known size, as mentioned in method 6, is by far the most direct and versatile approach and has found widespread acceptance. Reference blocks can be used to standardize both contact and immersion tests. To be most effective, however, it is obviously important that all reference blocks containing artificial discontinuities of identical size and location in the block exhibit the same ultrasonic response for a given set of test parameters.

Early Reference Blocks

As applications of ultrasonic flaw detection increased, a wide variety of reference blocks containing synthesized discontinuities were developed. Several of the more common early reference blocks are shown in Fig. 2. The first commercial instrument, the Reflectoscope, was supplied with a single 1 by 3 by 4-in. aluminum test block containing three very small drilled holes to simulate various flaw sizes. To date, about 1500 Sperry test blocks of this type have been distributed. While intended primarily for instrument setup purposes and for a rough check on operation, they have been used occasionally as standards for setting material acceptance limits.

These early Sperry setup blocks were arranged to provide reflection of the ultrasonic beam from the cylindrical surface of the hole formed by drilling into the 3 by 4-in. face of the block. Although these blocks appeared adequate for certain qualitative ultrasonic tests, a trend soon developed to employ the end surface of a flat-bottomed hole as a reflecting discontinuity of known shape and area. Aside from providing greater versatility in artificial discontinuity size, it was the consensus that the flat-bottomed reference holes more closely resembled several types of actual metallurgical discontinuities and that their use would promote more accuracy in establishing acceptance limits. Several early applications of blocks containing flat-ended holes are cited in the bibliography.

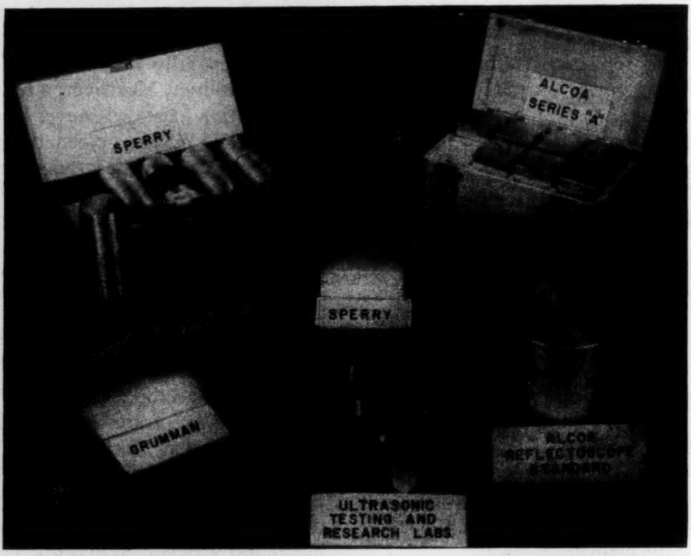

SPERRY
ALCOA SERIES "A"
SPERRY
GRUMMAN
ALCOA REFLECTOSCOPE STANDARD
ULTRASONIC TESTING AND RESEARCH LABS

FIG. 2.—Several Early Types of Ultrasonic Standard Reference Blocks.

In 1949, the Grumman Aircraft Engineering Corp. began producing a series of multiple-hole aluminum blocks. A typical Grumman type block, illustrated in Fig. 2, was 1½ by 6 by 15 in. and contained a number of flat-bottomed holes drilled into the 1½ by 15-in. face and spaced 1½ in. apart; the size of the holes ranged from $\frac{1}{64}$ to $\frac{3}{8}$ in. in diameter. The Grumman blocks led to the first proposed ASTM multipurpose reference block in 1951.

During this period, the Alcoa Research Laboratories began investigational work to develop a suitable Reflectoscope standard. This work resulted in the fabrication of a limited number of blocks of the type illustrated in Fig. 2. The Alcoa Research Laboratories subsequently cooperated with the airframe industry to develop a series of blocks, each containing only one flat-bottomed hole, thereby eliminating the possibility of interference from adjacent holes. A set of blocks referred to as the Alcoa Series A Ultrasonic Standard Reference Blocks, also shown in Fig. 2, was then established. This set consisted of eight 2 by 2 by $3\frac{3}{4}$-in. 2014-F aluminum alloy blocks; each block contained a flat-bottomed test hole drilled $\frac{3}{4}$ in. deep and centered in one 2 by 2-in. face. The test holes ranged in size from $\frac{1}{64}$ to $\frac{8}{64}$ in. in diameter, in $\frac{1}{64}$ in. diameter increments, and were numbered accordingly for identification. As a service to the industry, about 165 sets of Series A reference blocks were distributed at cost.

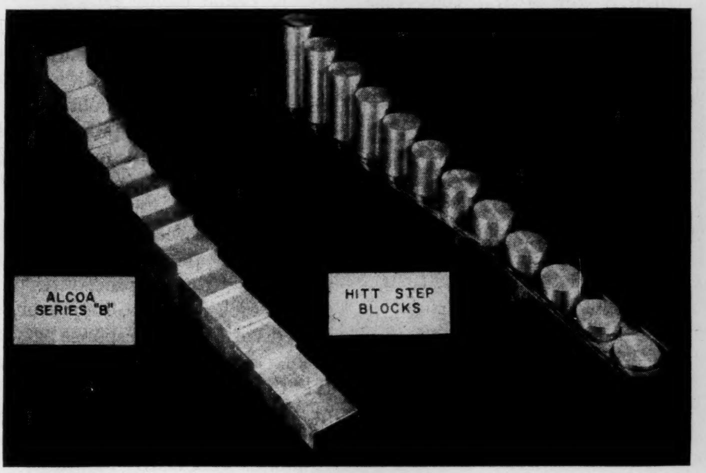


Fig. 3.—Distance-Amplitude Reference Blocks Used Commercially to Establish Acceptance Limits for Aluminum Alloy Products.

A number of interested organizations, including the Alcoa Research Laboratories, Sperry Products, Inc., and the Ultrasonic Testing and Research Laboratories, continued investigational programs relating to standardization problems. The subsequent development of more refined inspection techniques such as immersed testing seemed to require an expanded line of test blocks providing not only a range of test hole sizes but also a number of metal distances. Upon the introduction of the type UW Reflectoscope, Sperry Products began distributing a set of eight step blocks, shown in Fig. 2, with each instrument. The primary purpose of this Sperry set was to provide a means for checking the performance of the equipment and not necessarily to establish material acceptance limits.

A more complete set of step blocks containing twelve metal distances ranging from $\frac{1}{8}$ to $6\frac{1}{4}$ in. for each of three hole sizes ($\frac{3}{64}$, $\frac{5}{64}$, and $\frac{8}{64}$ in. diameters) were made available by the Ultrasonic Testing and Research Laboratories (Van Nuys, Calif.) in a number of materials and alloys. In expanding its service to the industry, Alcoa supplied similar 2014-F step blocks which were referred to as Alcoa Series B Ultrasonic Standard Reference Blocks. A complete set of B blocks contained 36 blocks and provided 12 metal distances ranging from $\frac{1}{8}$ to 6 in. for each of three hole sizes ($\frac{3}{64}$, $\frac{5}{64}$, and $\frac{8}{64}$ in. diameters). The Ultrasonic Testing and Research blocks as well as the Alcoa B blocks are shown in Fig. 3. It is estimated that a total of 100 sets of Hitt Aluminum alloy step blocks and 65 sets of Alcoa series B blocks were produced and distributed during the 1953–1958 period prior to the formulation of the approved ASTM recommended practice.

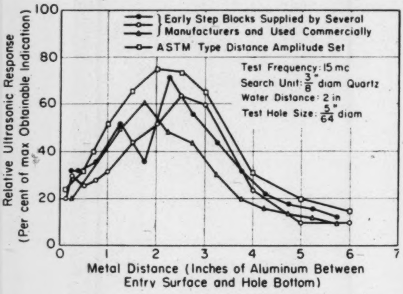


FIG. 4.—Comparison of Distance-Amplitude Curves Obtained by the Immersion Method from Different Sets of Commercial Step Blocks Containing Test Holes of the Same Size.

Unlike the earlier reference blocks which were used primarily to establish instrument sensitivity, the step blocks were employed as comparison standards. Acceptance of an inspected part was based on comparison between the indication height from a detected discontinuity located at some depth in the part and the indication height from a reference block containing a flat-bottomed hole of a specified size at a metal distance approximately equal to the depth of the discontinuity in the part. This practice, which is now accepted as standard in the aluminum and aircraft industries, partially compensates for the effect of metal distance or location in depth upon the ultrasonic response from a discontinuity. In view of this application, the distance-amplitude characteristics of reference blocks became important. These characteristics are usually defined by curves showing the ultrasonic response from test holes of constant size as a function of metal distance for a given set of step, or distance-amplitude, blocks.

While the early reference blocks were fabricated with great care and were thoroughly tested before release, there were no industry-wide standards for checking reference blocks. As a result, variations in distance-amplitude characteristics were experienced, particularly when a set of blocks from one supplier was compared with a similar set obtained from another source using the same test parameters. These variations apparently were due to differences between suppliers in machining practices, physical tolerances, and the ultrasonic testing procedures used to check block response. Moreover, the observed variation in response between sets of blocks appeared to be exaggerated as the test frequency was increased. For example, when several sets were compared at a frequency of 2.25 Mc, their responses were nearly identical, but comparison between the same sets at 15 Mc exhibited substantial differences. A typical example of the degree of variation between several sets of blocks is illustrated in Fig. 4 which shows distance-amplitude curves obtained by the immersion method at 15 Mc from a number of sets using the same test instrument and search unit. The early step blocks shown in Fig. 3 were checked at frequencies of either 5 or 10 Mc, and reasonable uniformity was realized, at least between blocks produced by any one supplier. However, it was possible that these same blocks could exhibit enough variation at test frequencies above 10 Mc to preclude their use as suitable acceptance standards.

REQUIREMENTS FOR ASTM BLOCKS

Although a task group of Subcommittee VI had been actively attempting to formulate ultrasonic reference standards since its organization in 1951, no agreement regarding the type, size, and number of blocks required was established. As a result of subsequent experience with the early reference blocks used to standardize commercial inspection of aluminum alloy products, variations in the ultrasonic responses of these early blocks were recognized. However, at that time, no technique could be envisioned to permit calibration of blocks against a primary reproducible and industry-wide standard.

By early 1956, the results of investigational work conducted by interested organizations as well as the experiences resulting from applications of the early blocks were accumulated and included in a review of requirements for suitable ASTM aluminum alloy standard reference blocks. During this review, which initiated the most recent investigational work of the task group, consideration was given to (1) establishing several possible applications that essentially determined the type, size, and number of blocks in a proposed ASTM set; (2) investigating the physical characteristics of the proposed block in order to establish realistic machining tolerances; and (3) developing a suitable fundamental standard and a procedure for checking quality of stock as well as the ultrasonic response of reference blocks.

In reviewing block requirements, it was agreed that the primary application of aluminum alloy reference blocks was the standardization and control of the somewhat critical ultrasonic tests which are currently employed in the inspection of aluminum alloy products. The more critical tests also required a better definition of the performance characteristics of test equipment. It was felt that reference blocks also could be applied advantageously to determine such characteristics as dynamic range (ability to discriminate small changes in discontinuity size over a wide range), resolution (ability to detect discontinuities located just below the entry surface), and sensitivity (ability to detect discontinuities of very small size). Although the application of reference blocks to establish acceptance limits was considered important, the use of blocks to establish instrument characteristics was given preference because fewer blocks appeared necessary to establish equipment performance than the relatively large number of blocks required for establishing acceptance limits. However, to obtain maximum versatility, equal attention was given to both applications when a basic set of ASTM reference blocks was proposed.

In the interests of portability and economy and to encourage general adoption, the task group subsequently agreed to limit the number of blocks in the proposed ASTM basic set to no more than a dozen relatively small blocks. The blocks in the basic set were selected to provide a suitable range of metal distances and hole sizes considered necessary to obtain a maximum amount of information regarding distance-amplitude and area-amplitude relationships. The distance-amplitude relationships are used to set acceptance limits while both distance and area responses are required to check equipment performance. Since it was necessary to establish blocks which would be satisfactory for most pulse-echo ultrasonic tests by either the contact or the immersion method, care was exercised in selecting the basic set to consider as many conditions as possible in the range of those encountered in general purpose inspection. This range encompasses test frequencies of from 1 to 15 Mc, search unit sizes of up to $1\frac{1}{8}$-in. diameter, and several commercially available test instruments. Further considerations were directed toward establishing the range of ultrasonic signal amplitudes required to standardize commercial tests for quality assurance.

Several years' experience with the early step blocks in the aluminum and airframe industries had shown that the ultrasonic indications obtained from hole sizes of $\frac{1}{64}$ to $\frac{8}{64}$ in. diameter were adequate for defining quality of aluminum alloy wrought products. In fact, many existing ultrasonic testing specifications require only three hole diameters ($\frac{3}{64}$, $\frac{5}{64}$, and $\frac{8}{64}$ in.) and a number of metal distances for satisfactory definition of internal quality. Because the aforementioned sizes of blocks appeared to be satisfactory throughout the industry, the same hole sizes and metal distances were adopted for use in the ASTM basic set. Moreover, it was reasonable to assume that tests to check equipment performance would be most informative if they were standardized to the same blocks used to establish acceptance limits. Therefore the test hole sizes and block lengths included in the basic set were selected from similar blocks now used as standards. The recommended basic set of ten reference blocks is shown in Table I. Subsequent to establishing the above hole sizes, metal distances, and number of blocks in the proposed basic set, the task group directed its attention toward the design of the ASTM reference block. In addition to selecting an appropriate aluminum alloy to be used for the reference block, consideration was given to its general shape or geometry.

TABLE I.—RECOMMENDED BASIC SET OF REFERENCE BLOCKS.

Metal Distance, in.	Test Hole Diameter, in.		
	3/64	5/64	8/64
1/8	...	X	...
1/4	...	X	...
1/2	...	X	...
3/4	...	X	...
1 1/2	...	X	...
3	X	X	X
6	...	X	X

The selection of a suitable aluminum alloy was influenced by several minimum requirements. First, it was agreed that the material selected for blocks should exhibit a low and reasonably uniform attenuation factor. Second, the material should be free of significant internal flaws, and it must produce an acoustic noise level (hash) significantly lower than the indication from the smallest possible hole size to be considered as an ASTM standard ($\frac{1}{64}$ in. diameter). As a result of a substantial amount of work regarding the effect of attenuation upon the ultrasonic response from blocks, several investigators reported that no significant differences in response due to attenuation were noted in blocks fabricated from a number of the aluminum alloys commonly used in the airframe industry. Generally, the attenuation factors of most aluminum alloy wrought products appeared to be low and uniform, at least for test frequencies up to and including 15 Mc. These encouraging results implied that blocks fabricated from one alloy could be used effectively to establish acceptance for a wide variety of aluminum alloy products.

In regard to acoustic noise level, investigational work as well as experience in commercial testing revealed that the "hash" level was significantly lower when the ultrasonic beam was directed through an aluminum alloy product such as rolled bar in a direction parallel to the direction of metal flow than when the beam was directed normal to the direction of metal flow in the same material. Moreover, a relationship between the degree of working received by a rolled product and the acoustic noise level appeared to exist. For these reasons, the material selected for ASTM blocks was 7075-T6 aluminum-alloy, 2 to 2¼-in. diameter rolled bar. This aluminum-alloy rolled bar produced a relatively low acoustic noise level in both the longitudinal and transverse directions; availability as a common aluminum alloy product and good machining characteristics also influenced the choice made by the task group. The diameter of the stock was restricted to a somewhat limited range to insure a uniform acoustic noise level in all reference blocks to be produced in accordance with ASTM practices. Since the quality of the stock could affect the ultrasonic response of reference blocks, a standard ultrasonic test was devised to insure uniform, flaw-free material exhibiting a specified acoustic noise level.

Several practical considerations influenced the design of the proposed ASTM reference block which was established as a cylindrical block, 2 in. in diameter, containing one flat-bottomed hole drilled to a depth of ¾ in. into one end along the longitudinal axis of the block. It was the consensus that the cylindrical shape would facilitate economical machining and precise drilling practices. The 2-in. dimension was chosen to accommodate contact testing and to minimize the possibility of obtaining extraneous indications from the cylindrical sides of the blocks, particularly those of longer lengths. The axis of the hole was oriented parallel to the rolling direction of the stock so that little or no hash level would be experienced when indications from the hole bottom were obtained. Because ¾ in. is considered to be the maximum practical depth for drilling holes of very small diameter and because no difficulty was experienced previously with early reference blocks containing holes drilled to this depth, it was decided to maintain the ¾-in. depth in the design of the ASTM block. Finally, consideration was given to a suitable method of sealing the test hole to preserve the ultrasonic response from the block. It was decided to seal the block with a permanent metallic plug driven into a flat counterbore centered over the hole; this sealing procedure was proved to be superior to using various nonmetallic sealing materials such as those employed in early blocks.

CONTROL OF PHYSICAL CHARACTERISTICS

Subsequent to establishing the aforementioned general requirements for a suitable reference block, investigational work was initiated to determine the effect of specific physical characteristics upon the ultrasonic response from a block. The physical variables investigated included the roughness, flatness, and alignment of the entry surface, the back surface, and the flat end of the test hole. The purpose of evaluating the effect of these variables was to establish fabricating procedures and tolerances necessary to insure reproducibility of ultrasonic response.

Since ultrasonic waves must travel through the top surface of the block twice to obtain an indication from the hole bottom, it is necessary to standardize the characteristics of this entry surface if its effect upon the height of indication is to be minimized. Differences in surface roughness and flatness, as well as the type of surface as determined by the shape of the machining tool, could contribute to differences between reference blocks, particularly when test frequencies equal to or greater than 15 Mc are employed. Actually, a smooth polished entry surface is an ideal boundary for tests by the immersion method, but it is difficult to maintain satisfactory coupling during contact tests on very smooth surfaces. For this reason, it was necessary to establish an entry surface roughness that would provide satisfactory tests by both the contact and immersion methods.

To determine the effect of entry surface roughness upon ultrasonic response, a number of reference blocks of the same length and hole size were precisely fabricated from selected 7075-T6 aluminum alloy rolled rod. Initially, the blocks were polished to obtain nearly smooth surfaces and were checked to insure identical surface alignment and flatness. Ultrasonic indications were first obtained by the immersion method at all common frequencies (2.25 to 15 Mc) from each of the blocks, which were then subjected to additional machining. A very small amount of metal was removed from the entry surface of each block. Various cutting speeds were employed with each of three cutting tools (pointed edge, rounded edge, and grinding wheel) to obtain a range of roughness (5 to 110 microinches, rms, as measured by a profilometer) for each surface profile. Each block containing the remachined surface was again checked for alignment and flatness which were held constant. Another set of ultrasonic indications was obtained from the remachined blocks using the same test parameters employed in the initial ultrasonic tests. The ultrasonic test results were then compared.

Results of this work indicated that the ultrasonic response from the test holes decreased with increasing surface roughness. Although no significant change in height of indication due to increasing surface roughness (up to about 110 microinches, rms) was noted at 2.25 Mc, a slight decrease was noted at 5 Mc, and a greater decrease was experienced at 10 Mc. For a surface roughness of 110 microinches the 15-Mc response decreased about 25 per cent from the response obtained with a smooth surface. At this same roughness, the height of indication decreased 4 and 12 per cent for frequencies of 5 and 10 Mc, respectively. However, for surface roughness less than about 50 microinches no loss in response at any test frequency was experienced. The surface profile appeared to have no effect upon response in the range of roughness up to 50 microinches. Above this range, the surface profile exhibited a slight effect: the least variation in response was experienced with the ground surface, followed by the surface obtained with a rounded tool, and finally, by the V-shaped surface which caused the greatest loss in response. The maximum decrease in response due to surface profile alone, however, was only about 4 per cent.

In view of these results, it was decided to establish the entry surface roughness of the ASTM block at 25 microinches, rms; this value provides a satisfactory surface for contact tests as well as for immersion tests at frequencies up to and including 15 Mc. To insure uniformity between blocks and to provide practical fabricating procedures, a tolerance of ±5 microinches, rms, was included, and a procedure for checking surface roughness was prescribed in the recommended practice. Since the results indicated that the surface profile exhibited negligible effect in the 20 to 30 microinch range, the type of cut to be used for preparing the entry surface was not specified. Moreover, the roughness of other surfaces of the block are not critical, and a standard roughness of 63 microinches, or smoother, was suggested primarily to maintain good appearance.

Similar tests were conducted by maintaining the entry surface roughness constant with varying surface flatness to determine its effect upon ultrasonic response. Results of this work indicated that a decrease in ultrasonic response of about 4 per cent might be experienced at 15 Mc if the entry surface was not flat within about 0.0005 in. This decrease became less significant as frequency was decreased below 15 Mc. Moreover, the loss was more apparent with a concave entry surface than with a convex entry surface. These results led to the 0.0002-in. tolerance for flatness now specified in the recommended practice.

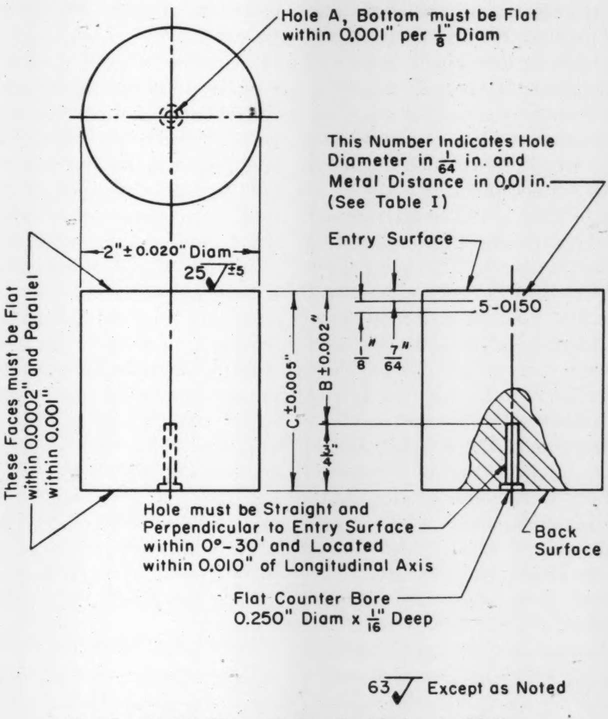

Hole A, Bottom must be Flat within 0.001" per $\frac{1}{8}$" Diam
This Number Indicates Hole Diameter in $\frac{1}{64}$ in. and Metal Distance in 0.01 in. (See Table I)
Entry Surface
2" ± 0.020" Diam
25 ±5
These Faces must be Flat within 0.0002" and Parallel within 0.001"
5-0150
$\frac{1}{8}$"
$\frac{7}{64}$"
C ± 0.005"
B ± 0.002"
$\frac{3}{4}$"
Hole must be Straight and Perpendicular to Entry Surface within 0°-30' and Located within 0.010" of Longitudinal Axis
Back Surface
Flat Counter Bore 0.250" Diam x $\frac{1}{16}$" Deep
63 Except as Noted

Fig. 5.—ASTM Ultrasonic Standard Reference Block.

The alignment of the hole bottom with respect to the entry surface is another critical factor which could limit the reliability of reference blocks as adequate standards. Although misalignment of the flat end of the test hole can be corrected for in immersion tests by angulating the search unit with respect to the entry surface, it would be difficult to obtain a maximum indication by the contact method, and an erroneous ultrasonic response would be encountered. Moreover, error in response due to improper orientation of the hole bottom usually increases with increasing test frequency. Since the proposed standards must be usable for frequencies up to 15 Mc, exacting tolerances on alignment were obviously necessary.

In order to establish tolerances on hole alignment, it was first necessary to prescribe the allowable tolerance in alignment, or parallelism, between the entry surface and the back surface, because either one of these surfaces could be used as a reference base during the drilling operation. For all practical purposes, if the two ends of the block are maintained parallel within 0.001 in. no effect upon ultrasonic response will be evident. However, if the hole bottom is misaligned to an angle greater than 0.5 deg with the entry surface, a decrease in height of indication can be expected, particularly at 15 Mc. The establishment of this limit of 0.5 deg in orientation, therefore, was considered mandatory. To control fabricating practices, procedures for mechanically checking entry surface alignment as well as ultrasonically checking hole bottom alignment were also considered important and were subsequently included in the recommended practice.

At the conclusion of the investigational work regarding the effect and control of physical characteristics, fabricating specifications and tolerances were established as shown in Fig. 5. Specifications governing over-all length, metal distance, and hole size were determined primarily by practical consideration of best machining practices. Additional tolerances on block diameter, location of the test hole in the back surface, and location of identification numbers were included to insure uniformity in appearance.

Although precise machining procedures and accurate measurement of the external physical characteristics will minimize the effect of a number of variables, mechanical measurement alone cannot predict the final ultrasonic behavior of a newly fabricated block. Mechanical features of the test hole such as finished diameter, orientation with respect to the external surfaces, flatness and roughness of the hole bottom, and actual metal distance between the hole bottom and entry surface will determine the final ultrasonic response from the block. These features are difficult to measure directly, and, of necessity, must be checked ultrasonically to insure uniformity of response between blocks.

One approach used with early reference blocks involved comparing the indication from new blocks to the indication from similar blocks retained as masters for this purpose, and only blocks giving comparable indications within an established tolerance of indication height were accepted. However, this procedure restricted uniformity of response to blocks fabricated only by one supplier. Unless other suppliers of blocks used identical master sets, a wide range of ultrasonic response from identical sets of reference blocks (see Fig. 4) was experienced. Moreover, master sets used as comparison standards were subject to aging, damage, corrosion, or loss, and, as a result, the ultrasonic characteristics of the master set itself could change.

In view of this, it was recognized that a primary standard was necessary to eliminate the need for a master set and to insure uniformity between all blocks manufactured and tested by any competent ultrasonic inspection laboratory in accordance with ASTM recommended practices. The work of the task group was subsequently directed toward investigating and developing a suitable primary reference standard and ultrasonic testing procedure to be used for checking the ultrasonic characteristics of test holes in reference blocks.

PRIMARY REFERENCE STANDARD

Results of several previous investigations concerning block characteristics indicated that tests by the immersion method were necessary to determine adequately the ultrasonic behavior of reference blocks. It was also found that tests at 15 Mc using a $\frac{3}{8}$-in. diameter quartz immersion search unit were more critical in revealing important differences between blocks than tests employing lower frequencies and search units of different size. Since several blocks in the basic set contain short metal distances of $\frac{1}{8}$ and $\frac{1}{4}$ in., adequate resolution such as that obtained with a test frequency of 15 Mc was necessary. Moreover, by requiring uniformity between blocks at this frequency, excellent performance at the more commonly used lower frequencies is assured. For these reasons, an ultrasonic test by the immersion method using a test frequency of 15 Mc was recommended for checking block response.

Precise checking of reference block response requires adjustment of test instrument sensitivity to a standard reproducible level prior to obtaining an indication from the hole bottom. However, it is difficult to maintain the instrument sensitivity characteristics at a constant level because changes in vacuum tube performance, internal voltages, and components may alter instrument performance at any time. One way to compensate for possible changes in equipment performance and to insure a constant sensitivity level is to adjust the gain of the instrument to obtain a predetermined magnitude of indication from some reflecting surface of specified size.

A promising technique involving the use of steel balls immersed in water to provide primary reference signals was developed and suggested to the task group. Since it appeared to offer several major advantages, this suggested technique was investigated to determine its merit as a possible standardization procedure to control the ultrasonic test recommended for checking reference block response. It was reasoned that if the reproducibility of this ultrasonic test could be assured by using ball reference standards, a direct comparison could be made between the indication from any given reference block and the indication from a ball of a specified size. This procedure would provide equivalent ball-block relationships for reference blocks of any length and hole size, and acceptable block response could be defined simply by a reference ball size and a water distance.

One advantage in using steel balls as primary references is realized in the wide range of sizes available commercially which insures the establishment of a suitable ball for any size reference block. Moreover, commercial steel balls of ball-bearing quality usually are precise in size and geometry, and uniform in surface finish and metallurgical structure. This contributes to uniformity of response because the aforementioned ball characteristics certainly determine the height of the ultrasonic indication obtained from its surface. Since a ball presents a spherical reflecting surface, obtaining a maximum signal for standardization purposes is simplified because angulation of the search unit is not critical, and the usual manipulation required to maximize the signal during immersed testing is not required.

Upon considering all the desirable aspects of using reference balls as primary standards, the problem of standardizing block checking tests is reduced to establishing suitable limits upon the few additional variables that affect the height of indication from the ball and the resulting accuracy of the standardization. These additional variables which were evaluated include the water distance between the crystal surface and the ball surface, the characteristics of the water used as a couplant, and the characteristics of the search unit employed in the test.

Since the distance between the crystal face and the reflecting surface in any transmitting medium is known to affect the amount of reflection obtained, it was necessary to investigate the effect of water distance upon the response from a ball in order to establish a fixed water distance to be used for the primary standard. Distance-amplitude curves were obtained by maximizing the indication from a steel ball at various increments of water distance. Because an ultrasonic test using a frequency of 15 Mc and a $\frac{3}{8}$ in. diameter quartz search unit was previously established to check reference blocks, investigations regarding the effect of distance were limited to these parameters. The apparatus used for this investigation included several commercial testing instruments exhibiting performance characteristics as prescribed in the recommended practice and standard manipulating equipment. Because it was evident that a wide range of balls would be necessary to check reference blocks, a range of ball sizes was included to determine the effect of size, if any, upon the distance-amplitude curve. Subsequent evaluation of test results indicated that the effect of instrumentation upon the ball response curve appeared negligible; this also was true for all ball sizes included.

A typical distance-amplitude curve showing the variation in response from a ball in water is shown in Fig. 6. As was expected, the curve exhibited several pronounced peaks (marked *A*, *B*, and *C* in Fig. 6) in the near-field zone. These peaks were followed by one final peak (*L*) which is commonly referred to as the close-field zone peak beyond which the variation in response decreases exponentially. A minimum variation in magnitude is evident at each peak, and it was feasible to establish a fixed water distance that would provide an indication at a particular peak to minimize error due to slight variation in water distance. Ideally, the true close-field zone peak would be the obvious selection for the standardization point because the beam pattern at this point contains only one major lobe. Unfortunately, a 15 Mc, $\frac{3}{8}$ in. diameter quartz search unit will produce a close-field zone peak at about $9\frac{1}{2}$ in. of water; the indication obtained at this water distance is very low and requires high instrument gain settings to obtain readable indications. The high gain settings would, of course, limit the range of blocks that could be checked. For this reason, it was necessary to establish the standardization point on another peak of higher magnitude occurring at 3 in. (*C*). It was recognized that, since the location in water distance of this peak as well as the others was dependent upon the area of

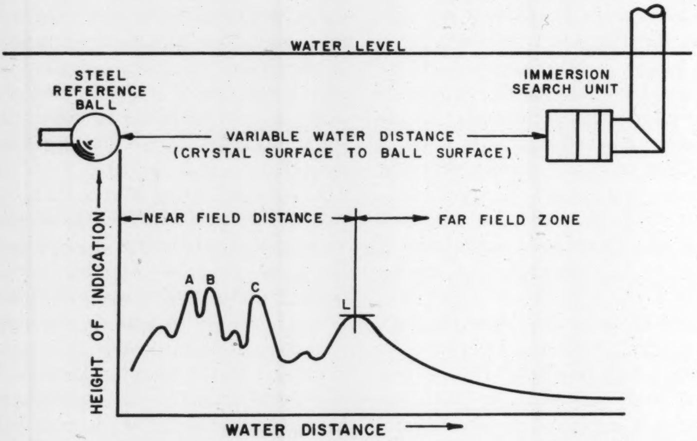


FIG. 6.—Typical Distance-Amplitude Response Curve Obtained from a Steel Reference Ball Immersed in Water.

the search unit, test frequency, and velocity of propagation, additional evaluations were necessary to determine the effect of these variables upon the location of the peaks selected for standardization. Obviously, a substantial shift in this peak could destroy the accuracy required for checking blocks.

Subsequent investigations were conducted to evaluate the influence of factors such as type of water, water temperature, and the use of deaerating and inhibiting agents upon the velocity of propagation which, in turn, affects generally the shape of the curve and particularly affects the location of the peak selected for the standardization point. Results of this work indicated that although all the aforementioned factors could change the position of the standardization peak, their effect could be minimized if the same test conditions were used for obtaining indications from both the reference ball and the block. The use of inhibitors appeared permissible provided they did not substantially alter the distance-amplitude characteristics of the reference ball in water.

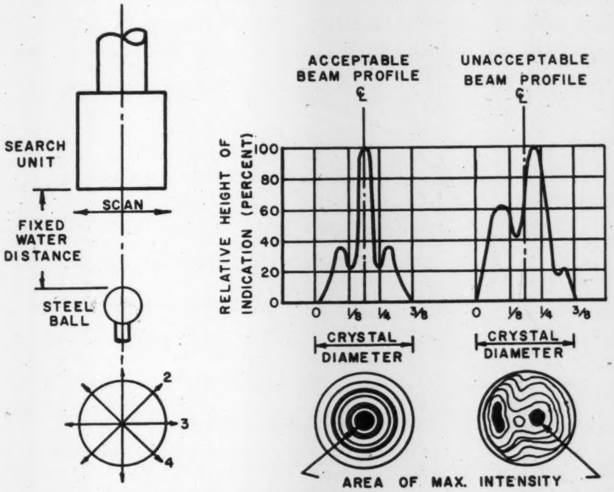


Fig. 7.—Diagrammatic Sketch Illustrating the General Procedure for Checking Beam Characteristics of 15 Mc, $\frac{3}{8}$ in. Diameter Quartz Search Units to be Used for Calibrating Reference Blocks; Typical Differences Between Characteristics of Various Search Units of the Same Type Are Included.

Subsequent to this initial work, it appeared that the only remaining variables that would affect the ultrasonic response from a ball at the established 3-in. water distance would be associated with the characteristics of the search unit used. As was previously mentioned, the location of the peaks in the curve depend upon the active area of the search unit. Experience with several types of commercial 15-Mc search units of the same nominal area indicated that differences in effective area existed. Obviously, unless some control was placed upon the characteristics of search units used for checking blocks, the desired uniformity of block response would not be realized.

In view of this, a number of 15-Mc search units were evaluated to determine the degree of variation in response that might be expected. Of a total number of ten units included in this work, only four units exhibited nearly identical distance-amplitude curves from balls in water. The primary difference noted between the curves obtained with the remaining units and those obtained with the four identical units was the change in location of the peaks. Subsequent attempts to use all of these units for establishing ball-block comparisons exhibited enough differences between the two groups of units to necessitate selecting search units to be used in the recommended practices. Identical ball-block comparisons were possible with the four units exhibiting identical distance-amplitude characteristics, but differences in ball-block comparisons were noted with the remaining group of units.

Further investigation of the beam characteristics of these units revealed differences in beam symmetry between two groups. It was found that the search units which gave uniform ball-block comparisons produced symmetrical beam profiles, whereas the other units that caused erroneous ball-block comparison exhibited unsymmetrical beam characteristics. The typical degree of variation between acceptable and unacceptable units is illustrated in Fig. 7.

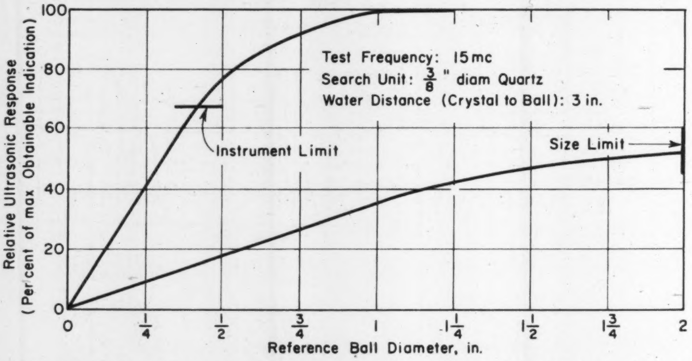


FIG. 8.—Area-Amplitude Response Curves from Various Sized Steel Balls in Water Showing Limitations in the Primary Reference Standard Due to Instrument Nonlinearity and Increasing Ball Size.

The beam characteristics of a search unit can be adequately defined by (1) obtaining a distance-amplitude curve from a ball of specified size in water and (2) determining the beam profile characteristics of the search unit as a ball is traversed diametrically across its face at a fixed water distance. The general scheme for obtaining distance-amplitude characteristics is shown in Fig. 6; beam characteristics are obtained by the procedure illustrated in Fig. 7. Adequate determination of beam symmetry requires at least four diametrical scans across the face of the crystal, each displaced to an angle of 45 deg, as shown.

Since the search unit characteristics are very important in maintaining uniformity, tests to determine the distance-amplitude characteristics as well as beam profile characteristics were specified, and only search units exhibiting specified performance characteristics were considered acceptable for checking blocks.

BALL-BLOCK COMPARISONS

Because a wide variety of lengths and hole sizes were included in the basic set, it was evident that a wide range of sensitivity levels and ball sizes would be necessary to provide primary references for all the blocks that might be included. Area-amplitude response curves were obtained from balls of various sizes to obtain data describing the range of available sensitivities using the test parameters previously established. One area-amplitude curve was obtained at a low gain setting of the test instrument while another curve was obtained from the same balls at a very high instrument gain setting. These curves illustrating the range of usable ball response are shown in Fig. 8.

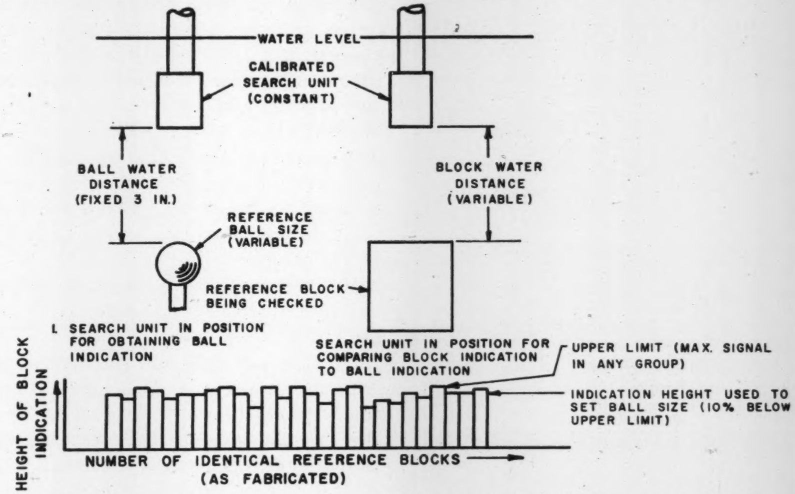

WATER LEVEL
CALIBRATED SEARCH UNIT (CONSTANT)
BALL WATER DISTANCE (FIXED 3 IN.)
BLOCK WATER DISTANCE (VARIABLE)
REFERENCE BALL SIZE (VARIABLE)
REFERENCE BLOCK BEING CHECKED
SEARCH UNIT IN POSITION FOR OBTAINING BALL INDICATION
SEARCH UNIT IN POSITION FOR COMPARING BLOCK INDICATION TO BALL INDICATION
UPPER LIMIT (MAX. SIGNAL IN ANY GROUP)
INDICATION HEIGHT USED TO SET BALL SIZE (10% BELOW UPPER LIMIT)
HEIGHT OF BLOCK INDICATION
NUMBER OF IDENTICAL REFERENCE BLOCKS (AS FABRICATED)

FIG. 9.—Diagrams Describing Procedure for Obtaining Initial Ball-Block Comparison Data and for Checking Ultrasonic Response from Blocks.

Maximum differentiation in signal height between balls as well as blocks is necessary to take care of small variations in block response adequately. However, to establish uniform response tolerances, a nearly linear instrument characteristic is desirable. The curve shown in Fig. 8 illustrates two limits of linearity. In the low gain curve, linearity becomes limited as ball size increases where the reflecting surface of a large ball begins to resemble a plane reflector of infinite size. In the range of large ball sizes, a slight increase in signal height will be experienced for a large change in ball diameter. For this reason, the ball sizes used to define acceptable block response were limited to a range below 2 in. in diameter.

The high gain limit in linearity is attributed directly to the amplifying characteristics of the receiver in the test instrument. No commercial ultrasonic testing instruments exhibit high gain linearity over their entire range. This is exemplified by the area-amplitude curves shown in Fig. 8, where departure from a straight line relationship occurs at an indication height slightly above 50 per cent of the maximum obtainable indication. To minimize instrument nonlinearity, the recommended practice specifies that the magnitude of indications employed for checking reference blocks be restricted to a range below 50 per cent of maximum.

Because it is very difficult to establish, theoretically, the correct response for a block of a given length and hole size, a more practical approach to establishing requirements of acceptable blocks was followed. It was reasoned that if a number of identical reference blocks were fabricated in accordance with the precise standards previously established, and if care was exercised to insure maximum ultrasonic response from each block, a realistic response level could be established statistically. A number of basic sets were fabricated using the best machining practices and maximum drilling precision in accordance with the proposed tolerances and specifications shown in Fig. 5. After thoroughly cleaning and mechanically checking each block, ultrasonic indications were obtained and compared for all blocks of the same size. A typical variation in ultrasonic response for a number of identical reference blocks is illustrated in Fig. 9. The maximum response in this group of blocks should represent a realistic maximum level. On the assumption that further improvement in machining practices would not produce blocks giving higher response, the highest signal amplitude shown was considered to be representative of a nearly ideal response.

TABLE II.—COMPARISON DATA FOR CHECKING ULTRASONIC RESPONSE OF BASIC SET.[a]

Block Identification Number	Hole Size, 1/64 in.	Metal Distance, in.	Block Water Distance (Crystal-to-Block Surface), in.	Equivalent Ball Diameter, in.	Ball Water Distance (Crystal-to-Ball Surface), in.
3-0300.....	3	3.000	3	$\frac{7}{16}$	3
5-0012.....	5	0.125	1	$\frac{15}{32}$	3
5-0025.....	5	0.250	1	$\frac{13}{16}$	3
5-0050.....	5	0.500	1	$\frac{31}{32}$	3
5-0075.....	5	0.750	1	1	3
5-0150.....	5	1.500	3	$1\frac{1}{8}$	3
5-0300.....	5	3.000	3	$1\frac{1}{2}$	3
5-0600.....	5	6.000	3	$\frac{7}{16}$	3
8-0300.....	8	3.000	6	$1\frac{1}{4}$	3
8-0600.....	8	6.000	3	$\frac{5}{8}$	3

[a] These data are tentative and subject to revision prior to final adoption.

Some time ago, it was decided by the task group that an ASTM block would be considered acceptable if its response was in a range of ±10 per cent of a specified value. To obtain an average value within the ±10 per cent established tolerance, the acceptable block indication was established as a value 10 per cent below the maximum signal shown in the bar chart of Fig. 9. When this height of indication was obtained, a ball was selected to give an ultrasonic response equivalent to it. A diagram illustrating the procedure for extablishing ball-block comparisons and for checking reference block response is also included in Fig. 9. The procedure for obtaining equivalent balls was repeated for each size of block included in the basic set and the ball-block comparison data shown in Table II was compiled.

In some cases, it was necessary to vary the water distance between the block and the crystal to keep the required ball size within the size limit and the instrument limit shown in Fig. 8. Although it is necessary to fix the water distance between the crystal and the ball, varying the water distance between the block and the crystal did not significantly affect accuracy.

EVALUATION OF RESULTS

The effectiveness of the completed recommended practice was subsequently evaluated through the cooperation of several members of the task group. Each cooperating member fabricated several basic sets of reference blocks in accordance with the proposed ASTM recommended practice. The blocks were then checked using the recommended procedures, first by the fabricator of the blocks and then by other members of the task group. Except for a few differences in block response, a favorable comparison between the block and the specified ball was obtained. The lower block response experienced with several of the blocks in the early skeleton sets was attributed either to minute scratches on the flat end of the test hole or to minute particles of dirt located at the bottom of the test hole. This experience further emphasized the need for precise drilling practices as well as thorough cleaning prior to checking block response.

A further evaluation of the results of the task group was made by obtaining distance-amplitude and area-amplitude response curves from about 35 basic sets which were subsequently produced. The combined characteristics of all the basic sets included in this work are shown in Fig. 10, which illustrates the range of variation which can be expected with ASTM basic sets. Except for a few isolated cases, all blocks gave indications within the limits described by the curves in Fig. 10. This 20 per cent scatter band exhibited by ASTM blocks represents a significant improvement over the re-

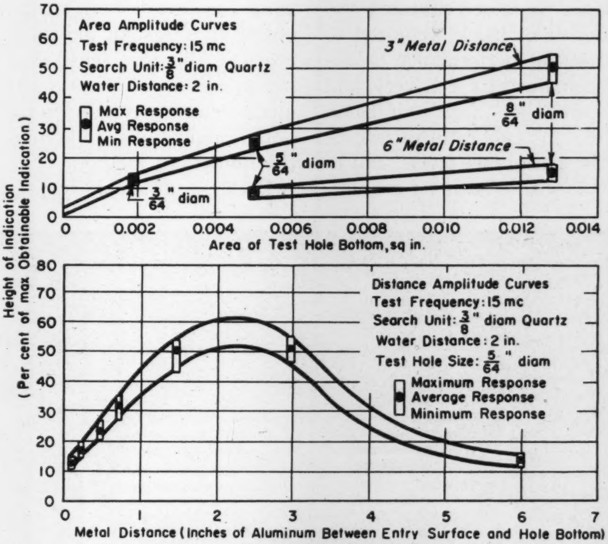


FIG. 10.—Area-Amplitude and Distance-Amplitude Curves Obtained by the Immersion Method from a Number of Basic Sets Showing the Range of Variation in Ultrasonic Response.

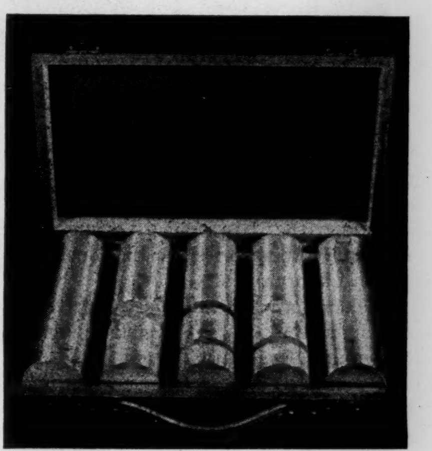


FIG. 11.—Basic Set of ASTM Ultrasonic Standard Reference Blocks Now Commercially Available.

sponse characteristics of earlier reference blocks shown in Fig. 4.

APPLICATIONS OF ASTM BLOCKS

The proposed basic set contains the minimum number of blocks required to establish equipment performance (sensitivity, resolution, linearity, recovery, and saturation), to check search unit beam characteristics, to calibrate ultrasonic tests to specific acceptance levels, and to serve as secondary standards for evaluating other test blocks. Basic sets such as those illustrated in Fig. 11 are now available commercially from several sources including the Ultrasonic Testing and Research Laboratories and the Alcoa Research Laboratories.

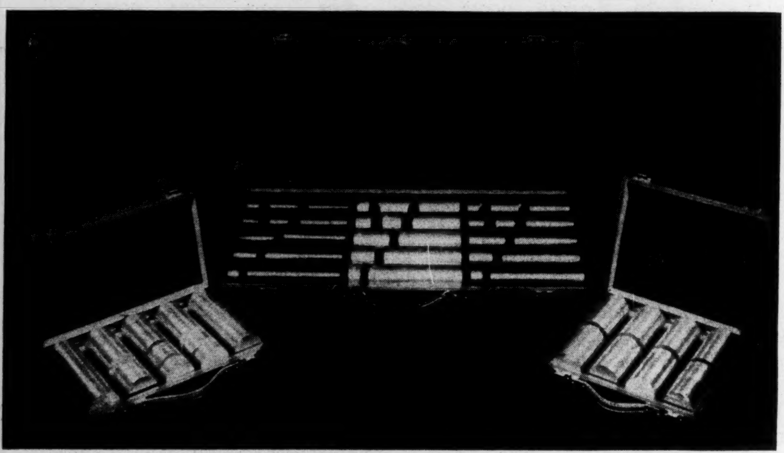


FIG. 12.—Proposed Sets of ASTM Ultrasonic Standard Reference Blocks. (*Left*) Basic set; (*Center*) distance-amplitude set; and (*Right*) area-amplitude set.

In considering the aforementioned uses of reference blocks, an obvious question concerning the feasibility of using the ball standards instead of blocks becomes apparent. Actually, the fundamental ball standards can be used in a number of these applications. Reference balls are suitable for the evaluation of several instrument performance characteristics such as receiver sensitivity, linearity, and saturation level. They are readily adaptable to determining the ultrasonic beam characteristics of search units, and their value has already been recognized in providing an ultrasonic indication for standardizing the test sensitivity of special immersion tests. Of necessity, however, ultrasonic tests employing ball targets to obtain indications are limited to tests by the immersion method.

Because ultrasonic inspection by the contact method requires direct contact of the search unit with the entry surface of a part, reference blocks are necessary for standardization. Moreover, correction of ultrasonic indications for the effect of distance requires the use of metallic reference blocks of the same base metal for either contact or immersion tests.

Frequently it is desirable to calibrate the time scale on the screen of a testing instrument to represent increments of depth in the part being tested. For this purpose it is convenient to use reference blocks of the same base metal as the part under inspection because the longitudinal velocities in both the block and the part are then essentially equal. Accurate depth calibration for aluminum may be difficult if water is employed as the only transmitting medium because of the substantial differences in velocity between water and aluminum.

Instrument performance characteristics such as resolution and recovery are affected by the magnitude of interface, or entry surface, interference. Therefore, these characteristics can only be determined with reference blocks which contain an entry surface. Obviously, the effect of ultrasonic energy passing from one medium to another through an entry surface cannot readily be established for a ball target immersed in water. The determination of contact search unit characteristics, in most cases, will require the use of reference blocks instead of a ball in water. Generally the reference ball standards may be applied to a number of tests, particularly those of an experimental nature performed in a laboratory, but reference blocks are also applicable to laboratory investigational work and are mandatory in controlling much routine ultrasonic inspection because of specification requirements.

STATUS

A substantial amount of work has already been done to establish ASTM requirements for a large number of blocks other than those included in the basic set. In fact, an expanded line of ASTM ultrasonic reference blocks such as those illustrated in Fig. 12 is already available. These sets provide important area-amplitude and distance-amplitude relationships similar to the relationships obtained with the Alcoa Series A blocks and the early step blocks. Additional committee work employing these blocks to develop procedures for checking ultrasonic equipment is now under consideration. The recommended procedures for checking beam characteristics of acceptable search units are also being considered for general standardization of search unit performance. When the results of this equipment standardization program are finalized, it is possible that improved equipment could again be applied to further improvement of the existing Recommended Practice for Fabricating and Checking Aluminum Alloy Ultrasonic Standard Reference Blocks (E 127 – 58 T).[3]

Acknowledgments:

The authors wish to thank the Aluminum Company of America, Sperry Products, Inc., and The Ultrasonic Testing and Research Laboratories, Inc., and, in particular, Dr. Wm. L. Fink, Alcoa Research Laboratories; Mr. J. C. Smack, Curtiss-Wright Corp.; Mr. W. C. Hitt of The Ultrasonic Testing and Research Laboratories; as well as all members of Task Group "B" of Subcommittee VI of E-7 for contributions to this program.

BIBLIOGRAPHY

(1) ASME Metals Engineering Design Handbook (O. J. Horger, Editor), Part IV-1, "Nondestructive Testing," Am. Soc. Mechanical Engrs., McGraw-Hill Book Co., Inc., New York, N. Y. (1953).

(2) ASTM Symposium on Ultrasonic Testing, Am. Soc. Testing Mats. (1949). (Issued as separate publication *ASTM STP No. 101.*) (Includes 350 references.)

(3) ASTM Symposium on Role of Nondestructive Testing in Economics of Production, Am. Soc. Testing Mats. (1950). (Issued as separate publication *ASTM STP No. 112.*)

(4) ASTM Symposium on Nondestructive Testing, Am. Soc. Testing Mats. (1952). (Issued as separate publication *ASTM STP No. 145.*)

(5) ASTM Symposium on Nondestructive

Testing, Am. Soc. Testing Mats. (1956). (Issued as separate publication *ASTM STP No. 213.*)

(6) ASTM Tentative Recommended Practice for Ultrasonic Testing by the Reflection Method Using Pulsed Longitudinal Waves Induced by Direct Contact (E 114 – 55 T), 1958 Book of ASTM Standards, Part 3.

(7) ASTM Tentative Recommended Practice for Fabricating and Checking Aluminum Alloy Ultrasonic Standard Reference Blocks (E 127 – 58 T), 1958 Book of ASTM Standards, Part 3.

(8) L. Bergmann, "Der Ultraschall und Seine Anwendung," in "Wissenschaft und Technik," S. Hirzel Verlag, Stuttgart (1954). (Includes 5000 references.)

(9) C. W. Cline and J. B. Morgan, "Standardization in Ultrasonic Testing," *Nondestructive Testing*, Vol. XIII, p. 23 (1955).

(10) W. L. Fink, "Ultrasonic Testing of Aluminum Alloys," ASTM BULLETIN, No. 226, Dec., 1957, p. 36 (TP224).

(11) F. A. Firestone, "Supersonic Reflectoscope for Interior Inspection," *Metal Progress*, Vol. 48, p. 503 (1945).

(12) H. D. Guinn, R. D. McKown, J. C. Folz and W. C. Hitt, "Research and Development Leading to the Establishment of Ultrasonic Test Standards for Aircraft Materials," WADC Technical Report 57-268 Oct., 1957.

(13) T. F. Hueter and R. H. Bolt, "Sonics," John Wiley & Sons, Inc., New York, N. Y. (1955).

(14) L. E. Kinsler and A. R. Frey, "Fundamentals of Acoustics," John Wiley & Sons Inc., New York, N. Y. (1950).

(15) R. E. Kleint, "Relationship Between Defect Orientation and Ultrasonic Inspection," *Nondestructive Testing*, Vol. XV, No. 1, p. 30 (1957).

(16) J. Krautkramer, "The Ultrasonic Field Pattern and Its Application to Materials Testing," *Zeitschrift für Metallkunde*, Vol. 48, No. 11, p. 606 (1957).

(17) W. P. Mason, "Piezoelectric Crystals and Their Application to Ultrasonics," D. Van Nostrand & Co., New York, N. Y. (1950).

(18) J. B. Morgan, "Some Factors of Importance in Ultrasonic Testing," *Nondestructive Testing*, Vol. XII, No. 3, May–June, 1954, p. 13.

(19) R. L. Roderick and R. Truell, "Measurement of Ultrasonic Attenuation in Solids by the Pulse Technique and Some Results in Steel," *Journal of Applied Physics*, Vol. 23, No. 2, Feb., 1952, p. 267.

(20) S. Serabian, "Influence of Geometry upon an Ultrasonic Defect—Size Determination in Large Rotor Forgings," *Nondestructive Testing*, Vol. XIV, p. 18, July–Aug., 1956.

(21) S. Serabian and G. E. Lockyer, "Ultrasonic Testing of Large Rotor Forgings—Criteria for Detecting Ability," ASME Paper No. 57-A-279, Dec., 1957.

(22) Rebecca H. Smith and D. C. Erdman, "Immersed Ultrasonic Inspection," *Iron Age*, Vol. 164, No. 5, p. 83 (1949).

(23) SNT Handbook of Nondestructive Testing —Ultrasonic Tests, Soc. Nondestructive Testing. (To be published.)

(24) SNT Airframe Committee, "Recommended Ultrasonic Acceptance Standards for Airframe Aluminum Alloy Plate, Forgings and Extrusions," *Nondestructive Testing*, Vol. XV, No. 6, p. 368 (1957).

(25) Sperry Products, Inc., Technical Proposal: TP-007, "Evaluation of Reference Blocks for Ultrasonic Inspection Standards," Response to P. R. 1116 (July, 1955) WADC-WCKCR.

(26) Ultrasonic Testing & Research Laboratory Reports, "Research and Development Leading to the Establishment of Ultrasonic Test Standards for Aircraft Materials," Contract AF 33(616)5877 (1958). (R. E. Kleint and R. D. McKown.)

(27) L. Van Ouwerkerk, "Relative Advantage and Limitations of Nondestructive Testing Methods," *Nondestructive Testing*, Vol. XV, No. 5, p. 298 (1957).

(28) H. E. Van Valkenburg, R. A. Wilson, and W. R. Olha, "Ultrasonic Inspection Equipment Program," Final Report TR-065, Air Material Command Contract AF 33(600)-29879 (1957).

(29) Z. J. Verney and R. E. Kleint, "SNT Airframe Committee Report on Reproducibility of Ultrasonic Inspection Test Results," *Nondestructive Testing*, Vol. XV, No. 5, p. 290 (1957).

(30) J. B. Morgan, "Ultrasonic Testing Procedures," *Nondestructive Testing*, Vol. XVII, No. 1, Jan-Feb., 1959, p. 32.

(31) J. Krautkramer, "Determination of the Size of Defects by the Ultrasonic Impulse Echo Method," *British Journal of Applied Physics*, Vol. 10, June 1959, p. 240.

(32) Jiro Yamanouchi, Noboru Takagi, and Noboru Niwa, "Sensitivity Standard Test Block for Ultrasonic Flaw Detector," *Journal of the Society for Nondestructive Inspection* (Japan), Vol. 4, No. 1, May 1955, p. 1.

DISCUSSION

Mr. Howard C. Skidmore.[1]—In Fig. 4 of the paper a series of curves was presented which described the variation between blocks manufactured by different companies. The water distance was 2 in. If the distance had been expanded to 3 in., would the curves have approached those indicated in the ASTM standard or would they still have been in that same shape?

Mr. F. C. Panian (*author*).—We have found that increasing the water distance from 2 to 3 in. does not change the general relationship of these curves. It should be noted that the curves shown in Fig. 4 were obtained at a test frequency of 15 Mc, while the early reference blocks were designed for tests at lower frequencies. As the test frequency is decreased, the differences between these curves tend to become less noticeable. The 15-Mc frequency was selected for checking reference blocks because it was found that this frequency appeared most critical in detecting differences between reference blocks.

Mr. Skidmore.—Has there been any difference noted in the response of a single or individual crystal with different circuitry? In other words, we have in the field several different manufacturers of instruments. Are the curves the same with the various instruments or do they change?

Mr. Panian.—We have found that the shape of the distance-amplitude curve from any given set of reference blocks will change when one search unit is used with several instruments. We also have found that a significant variation will be noted when distance-amplitude curves from one set of blocks are obtained with a number of similar search units and with the same instrument. Results of our work appear to indicate that the greatest variation in making ball-block comparisons is due to differences in search units. For this reason, it was necessary to specify the ultrasonic characteristics of the search unit to be used for checking blocks. However, it is generally agreed that a direct comparison between a number of sets of reference blocks using the same search unit and test instrument is a comparison on a somewhat arbitrary scale, and the same general relationship between sets of blocks usually will be obtained with any commercial instrument and with any search unit. The general shape of the curves may vary in this case, but their interrelationship will remain unchanged.

Mr. Skidmore.—There are several instruments on the market; one of them has a total maximum indication amplitude height of 2 in. Ten per cent of this, which is called for in the specification,[2] gets to be a fairly small quantity—in fact, close to the readable variations. Is this then a good criterion when you have a tolerance of 10 per cent?

Mr. Panian.—One of our problems

---

[1] Metallurgist, Quality Control Dept., Republic Aviation Corp., Farmingdale, N. Y.

[2] Recommended Practice for Fabricating and Checking Aluminum Alloy Ultrasonic Standard Reference Blocks (E 127 – 58 T), 1958 Book of ASTM Standards, Part 3.

in developing this specification was to eliminate the effect of the instrument as much as possible. I think you are referring to the effect of the instrument characteristics upon checking reference blocks. It will be noticed that, in the specification, we were careful to specify that reference blocks be checked in the linear range of the instrument. This range in indication amplitude is well below the 2-in. level to which you refer. Not much of an alternative is left except to read, as accurately as possible, the indication heights in this very small range of amplitudes.

MR. SKIDMORE.—If the initial instrument setting provides a 1-in. indication which is about 50 per cent of the maximum height as required by the specification, then 10 per cent is 0.1 in., and it is even harder to read that close.

MR. PANIAN.—I would like to emphasize that making and checking reference blocks in accordance with this specification is a critical job, and the problems involved in this work are not necessarily the same as those encountered in ordinary ultrasonic inspection. Because we are after something a little more critical, it must be realized that precise readings of indication heights while checking reference blocks is mandatory. It is unfortunate that the ultrasonic testing instruments of today limit the maximum height of readable indication to a range between 2 and 2½ in. Therefore precise measurement of changes in magnitude within the ±10 per cent range might be considered somewhat difficult.

CHAIRMAN A. H. KIDDER.[3]—As I understand it, the author is trying to outline the development behind the ASTM recommended practice for fabricating and checking ultrasonic standard reference blocks.

---

[3] Research Division, Philadelphia Electric Co., Philadelphia, Pa.

A COOPERATIVE STUDY OF THE HARDNESS TESTING OF CEMENTED CARBIDES*

By Betty M. Caugherty,[1] H. T. Oatman,[1] and O. W. Reen[1]

Synopsis

In an effort to obtain a standard testing procedure for determining the Rockwell hardness of cemented carbides, a cooperative test program has been conducted by the Cemented Carbide Producers Assn.

The data obtained from test blocks of varying hardness levels under given test conditions have been analyzed statistically and the range of variability reported. Possible reasons for the variability based on a study of the microstructures of the test blocks are listed.

Serious discrepancies in test data of cemented tungsten carbide between the producers and their customers as well as among the producers themselves have led to an intensive effort to standardize testing procedures for these very hard, brittle materials. Through ASTM Committee B-9, Subcommittee III-C on Cemented Carbides, the initial efforts were made to standardize testing procedures. Only a few test procedures have been standardized by this group.

The lack in standardized test procedures has been recognized by the Cemented Carbide Producers Assn., and its Technical Committee has undertaken the standardization of test procedures. Various test standards have been prepared and several have been submitted to the ASTM for consideration as ASTM standards.

Because of the general acceptance by both producer and consumer of the hardness test as a means of evaluating cemented carbides, the procedure for the determination of this property has received special attention. The Rockwell hardness test, because of its simplicity, speed of testing, and low capital investment, has become the generally accepted hardness test in the industry. Due to the hard, brittle character of the material, the hardness is measured with a Brale diamond indentor under a 60-kg load with the hardness indicated on the Rockwell A scale. Greater loads, such as the 150-kg load employed in the use of the C scale, create the problems of premature damage to the penetrator and cracking of the test specimen in the immediate area of testing.

In order to ascertain the best practice, each company member of the association submitted its testing procedure to the committee for evaluation. From these procedures, it was hoped that the best features of each could be incorporated into a single test procedure. A study of these techniques, however, revealed many wide variations in testing conditions such as sample preparation, anvil size, calibration methods, and operator technique.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Technical Statistician, Supervisor of Technical Data Center, and Staff Research Consultant, respectively, Allegheny Ludlum Steel Corp., Brackenridge, Pa.

While numerical values may be placed on minimum thickness and surface finish of the sample, the condition and inspection of the testing machine and the Brale diamond indenter, the physical location of the testing machine and operator technique can, at best, be described only qualitatively.

A set of six blocks at hardness levels varying between 84.5 and 93.0 on the Rockwell A scale was used for one round-robin test. The hardness testing of these blocks was conducted under prescribed testing conditions in which each machine was calibrated by the same calibrating blocks. In this manner, each machine was calibrated to the same hardness level, and the variability in results was due to other factors.

TABLE I.—TEST BLOCKS.

Test Block	Nominal Hardness, Rockwell A Scale	Composition, per cent by weight		
		Tungsten Carbide	Tantalum Carbide	Cobalt
A	84.5	68	5	27
C	85.5	80	...	20
D	87.0	84	...	16
F	89.0	87	...	13
B	91.0	93	...	7
E	93.0	97	...	3

In a second round robin test, a set of four blocks at hardness levels between 86.5 and 92.0 on the Rockwell A scale was tested in the quality control departments of the various companies. The machines used in this phase of testing were calibrated by the internal hardness calibrating blocks of the individual companies. The purpose of the test was to determine actual variability in hardness between companies when the usual methods of determining hardness were followed.

EXPERIMENTAL PROCEDURE

Cooperative Test Using Same Calibrating Blocks:

Materials.—Six test blocks were manufactured by conventional cemented carbide practice, with different hardness levels resulting from variations in grain size and analysis. The test blocks were produced by four carbide producing companies and had the nominal hardnesses and analyses given in Table I. The microstructures of these test blocks are shown in Figs. 2 to 7.

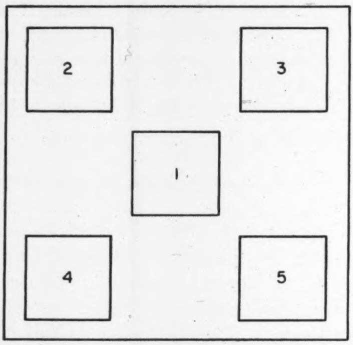


FIG. 1.—Test Areas of Cemented Carbide Calibrating and Test Blocks.

Preparation of Blocks.—Blocks were prepared in duplicate, one series designated as the "calibrating" blocks, the other designated as the "test" blocks. Blocks *A*, *B*, *C*, and *D* were 2 in. square; blocks *E* and *F* were $1\frac{1}{2}$ in. square. Both face areas of all the blocks were ground flat and parallel to a thickness of $\frac{1}{4}$ in. To insure freedom from the effect of surface irregularities, the blocks were ground to a surface finish of 15 microinches rms maximum.

Five $\frac{1}{2}$-in. squares were outlined by etching on all the calibrating and test blocks. The areas were assigned numbers, these numbers designating the testing

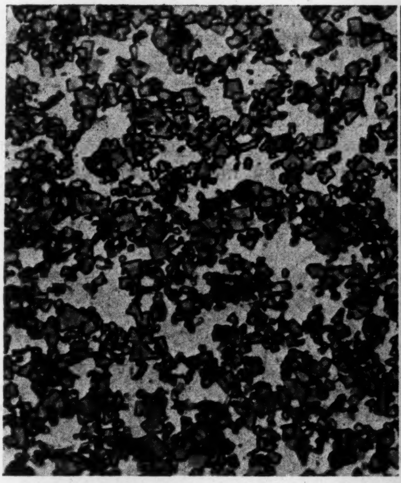


FIG. 2.—Test Block *A*, 68 per cent Tungsten Carbide, 5 per cent Tantalum Carbide, 27 per cent Cobalt; Rockwell Hardness A 84.7 Average. Etched in alkaline potassium ferricyanide (×1500).

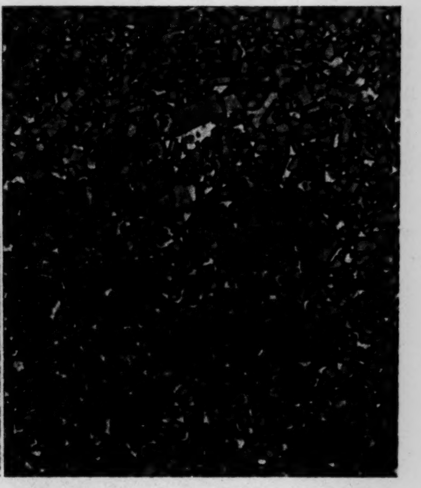


FIG. 3.—Test Block *B*, 93 per cent Tungsten Carbide, 7 per cent Cobalt; Rockwell Hardness A 91.0 Average. Etched in alkaline potassium ferricyanide (×1500).

FIG. 4.—Test Block *C*, 80 per cent Tungsten Carbide, 20 per cent Cobalt; Rockwell Hardness A 85.4 Average. Etched in alkaline potassium ferricyanide (×1500).

FIG. 5.—Test Block *D*, 84 per cent Tungsten Carbide, 16 per cent Cobalt; Rockwell Hardness A 87.0 Average. Etched in alkaline potassium ferricyanide (×1500).

area of the block. A typical layout of a block is sketched in Fig. 1.

Hardness Testing Procedure.—Hardness testing was conducted according to ASTM Methods E 18.[2] The major load application was applied in accordance with section 5 (*i*) (*1*) which states:

> "In the case of materials which do not exhibit plastic flow after application of the major load, the pointer will come to rest before the motion of the operating lever stops, and in this case the operating lever may be brought back to its latched position immediately after the pointer stops to reduce possible errors due to externally caused vibration."

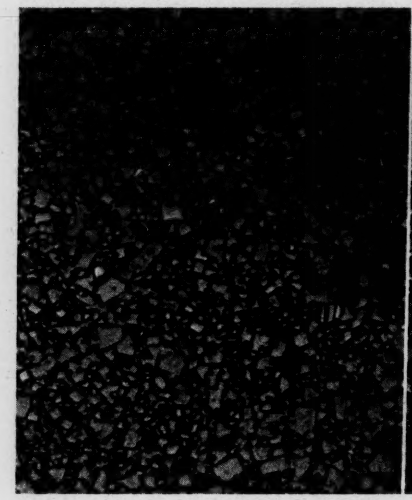


FIG. 6.—Test Block *E*, 97 per cent Tungsten Carbide, 3 per cent Cobalt; Rockwell Hardness A 93.1 Average. Etched in alkaline potassium ferricyanide (×1500).

FIG. 7.—Test Block *F*, 87 per cent Tungsten Carbide, 13 per cent Cobalt; Rockwell Hardness A 88.8 Average. Etched in alkaline potassium ferricyanide (×1500).

The machine manufacturer, type of Brale, and anvil size used by the various companies are listed in Table II. The main variation was in the anvil size where five companies used a $2\frac{1}{2}$-in. diameter anvil, the balance a smaller diameter, of the order of $\frac{5}{16}$-in.

Calibration of Machines.—The hardness instruments used in the test were calibrated by means of the calibrating blocks of the same order of magnitude as the test blocks. Hardness values were affixed to these blocks by one company using a machine standardized by their internal hardness standard. The calibrating blocks were sent with the test blocks to the participating companies, and the machines were calibrated at each hardness level just prior to the hardness determination of the test blocks. By calibrating each machine to some standard value, one variable was eliminated from the test procedure.

Determination of Data.—After calibrating the machine for the given hard-

---

[2] Tentative Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 57 T), 1958 Book of ASTM Standards, Part 3, p. 39.

TABLE II.—EQUIPMENT DATA AND ROCKWELL A HARDNESS OBTAINED IN COOPERATIVE HARDNESS TESTING OF CEMENTED CARBIDES.

Company	Machine Number	Diamond Brale Indenter Type	Brale Manufacturer	Type of Anvil, in.	Block A							Block B							Block C						
					Area No. 1	Area No. 2	Area No. 3	Area No. 4	Area No. 5	Correction Factor	Average of Reading	Area No. 1	Area No. 2	Area No. 3	Area No. 4	Area No. 5	Correction Factor	Average of Reading	Area No. 1	Area No. 2	Area No. 3	Area No. 4	Area No. 5	Correction Factor	Average of Reading
No. 1	3 JR	A	Wilson	0.310 diam.	84.5	84.7	84.5	84.8	84.8	0	84.7	91.0	90.9	91.0	90.8	91.2	0	91.0	85.2	85.3	85.2	85.3	85.4	0	85.3
No. 2	1 JR	A	Service Diamond	¼ face	83.8	84.2	84.3	84.3	84.5	−0.8	84.2	91.1	91.1	91.0	90.9	91.3	+0.2	91.1	85.2	85.3	85.4	85.2	85.5	−0.6	85.3
No. 3	3 JR	...	Wilson	0.310 diam.	84.5	84.5	84.6	84.7	85.0	−0.3	84.6	91.1	91.1	91.0	91.1	91.2	+0.2	91.1	85.3	85.3	85.3	85.6	85.5	0	85.4
No. 4	3 JR	C	Wilson	2½ diam.	83.1	81.4	82.6	84.0	84.5	+0.6	83.1	90.8	90.7	90.9	91.0	91.2	+0.6	90.9	85.8	85.1	85.8	85.3	85.0	+1.3	85.4
No. 5	1 JR	A	Wilson	2½ diam.	84.1	84.2	84.3	84.6	84.6	−0.6	84.4	91.2	91.1	91.1	91.1	91.2	−0.1	91.1	85.2	85.2	85.3	85.5	85.4	−0.6	85.3
No. 6	1 R	N	Wilson	⅝ diam.	84.2	84.3	84.3	84.2	84.6	+0.3	84.3	90.9	90.8	90.9	90.9	90.9	+0.7	90.9	85.4	85.2	85.4	85.4	85.4	+0.5	85.4
No. 7	3 JR	A	Service Diamond	0.310 diam.	84.4	84.5	84.4	84.4	84.4	−0.4	84.4	91.1	91.0	91.0	91.0	91.0	+0.2	91.0	85.4	85.5	85.5	85.6	85.6	−0.1	85.5
No. 8	1 JR	Conical	Wilson	2½ diam.	84.6	84.4	84.2	84.6	84.8	−0.1	84.5	91.1	91.0	91.0	91.1	91.1	+0.6	91.1	85.4	84.6	84.8	85.0	85.2	+0.1	85.0
No. 9	3 JR	A	Wilson	2½ diam.	84.1	84.1	84.1	84.1	84.3	−0.2	84.2	91.4	91.2	91.3	91.3	91.3	+0.3	91.3	85.3	85.4	85.3	85.5	85.5	0	85.4
No. 10	1 JR	A	Wilson	2½ diam.	84.4	84.6	84.5	84.6	84.8	−0.1	84.6	91.2	91.1	91.1	91.1	91.1	+0.3	91.1	85.3	85.3	85.2	85.5	85.4	+0.1	85.3
No. 11	1 JR	A	Wilson	0.310 diam.	85.2	85.0	85.0	85.2	85.2	−0.5	85.1	91.1	91.1	91.1	91.1	91.1	+0.1	91.1	85.3	85.3	85.4	85.3	85.4	−0.4	85.3
No. 12	3 JR	A	Wilson	0.310 diam.	84.4	84.6	84.3	84.7	84.7	−0.1	84.5	91.1	90.9	91.0	91.0	90.8	+0.1	90.8	85.4	85.3	85.4	85.4	85.3	+0.1	85.4
No. 13	3 YR	A	Wilson	0.312 diam.	84.4	84.4	84.4	84.5	84.5	−0.6	84.4	91.0	91.0	91.0	91.0	91.0	−0.2	91.0	85.4	85.3	85.4	85.3	85.3	−0.5	85.3
No. 14	1 JR	A	Wilson	0.312 diam.	84.3	84.2	84.3	84.6	84.6	−0.3	84.4	91.1	91.1	91.2	91.0	91.2	+0.4	91.1	85.5	85.4	85.2	85.7	85.6	+0.1	85.5

Company	Machine Number	Diamond Brale Indenter Type	Brale Manufacturer	Type of Anvil, in.	Block D							Block E							Block F						
					Area No. 1	Area No. 2	Area No. 3	Area No. 4	Area No. 5	Correction Factor	Average of Reading	Area No. 1	Area No. 2	Area No. 3	Area No. 4	Area No. 5	Correction Factor	Average of Reading	Area No. 1	Area No. 2	Area No. 3	Area No. 4	Area No. 5	Correction Factor	Average of Reading
No. 1	3 JR	A	Wilson	0.310 diam.	87.4	87.0	87.0	87.6	87.0	0	87.2	93.0	93.1	93.2	93.2	93.1	0	93.1	88.7	89.0	88.8	88.8	88.7	0	88.8
No. 2	1 JR	A	Service Diamond	¼ face	86.8	86.8	87.0	86.9	87.1	−0.4	86.9	93.0	93.0	93.2	93.2	93.1	+0.4	93.1	89.0	89.2	89.1	89.0	89.0	0	89.0
No. 3	3 JR	......	Wilson	0.310 diam.	87.0	87.1	86.8	87.2	87.0	0	87.0	93.3	93.2	93.0	93.1	93.4	+0.5	93.2	88.5	88.6	88.4	88.7	88.6	+0.1	88.6
No. 4	3 JR	C	Wilson	2½ diam.	87.4	87.3	87.2	87.5	87.4	+0.6	87.4	93.2	93.1	93.1	93.2	93.1	+0.7	93.1	88.7	89.0	88.9	89.0	88.8	+0.8	88.8
No. 5	1 JR	A	Wilson	2½ diam.	87.2	86.9	86.7	87.4	87.0	−0.5	87.0	93.2	93.1	93.3	93.3	93.3	+0.1	93.2	89.0	88.9	88.7	89.0	89.0	−0.2	88.9
No. 6	1 R	N	Wilson	⅝ diam.	87.3	87.3	86.9	87.3	86.9	+0.6	87.1	93.2	93.1	93.1	93.1	93.1	+0.9	93.1	88.8	88.8	88.7	88.8	88.8	+0.7	88.8
No. 7	3 JR	A	Service Diamond	0.310 diam.	87.0	87.1	86.7	87.0	86.7	−0.2	86.9	93.1	93.3	92.9	92.9	93.0	+0.5	93.2	88.9	88.9	89.0	88.9	88.9	+0.1	88.9
No. 8	1 JR	Conical	Wilson	2½ diam.	87.2	87.2	87.0	87.3	87.0	+0.3	87.1	93.1	93.0	93.0	92.9	93.0	+0.5	93.0	88.6	88.6	88.6	88.7	88.5	0	88.6
No. 9	3 JR	A	Wilson	2½ diam.	86.8	87.1	86.7	87.1	86.7	−0.1	86.9	93.1	93.0	93.0	93.0	93.0	+0.2	93.0	89.0	89.0	88.8	88.9	88.8	+0.1	88.9
No. 10	1 JR	A	Wilson	2½ diam.	86.9	86.9	86.7	87.0	86.8	−0.1	86.9	93.2	93.2	93.2	93.2	93.2	+0.5	93.2	88.9	88.8	88.8	89.0	89.0	+0.2	88.9
No. 11	1 JR	A	Wilson	0.310 diam.	87.1	87.2	86.9	87.5	87.0	−0.2	87.1	93.1	93.2	93.2	93.1	93.1	+0.2	93.1	88.9	88.8	88.8	88.8	88.9	0	88.8
No. 12	3 JR	A	Wilson	0.310 diam.	87.1	87.2	86.8	87.3	87.0	0	87.1	93.0	92.8	93.0	93.1	93.0	+0.2	93.0	88.7	88.8	88.8	88.7	88.8	0	88.8
No. 13	3 YR	A	Wilson	0.312 diam.	87.2	87.5	86.8	87.5	86.9	−0.3	87.2	93.1	93.2	93.1	93.3	93.2	0	93.2	88.7	88.7	88.6	88.7	88.6	−0.4	88.7
No. 14	1 JR	A	Wilson	0.312 diam.	87.0	87.1	86.7	87.3	86.8	−0.1	87.0	93.1	93.1	93.1	93.2	93.1	+0.5	93.1	88.9	88.7	88.5	88.6	88.5	+0.1	88.6

ness range, each company obtained hardness values on blocks *A* to *F* in the designated ½-in. square areas. The hardness values for each area were observed, corrected, and recorded. These results are shown in Table II.

TABLE III.—TEST BLOCKS.

Test Block	Nominal Hardness, Rockwell A Scale	Composition, per cent by weight	
		Tungsten Carbide	Cobalt
G............	86.0	88	12
H............	89.0	89	11
I............	91.0	94	6
J............	92.5	94	6

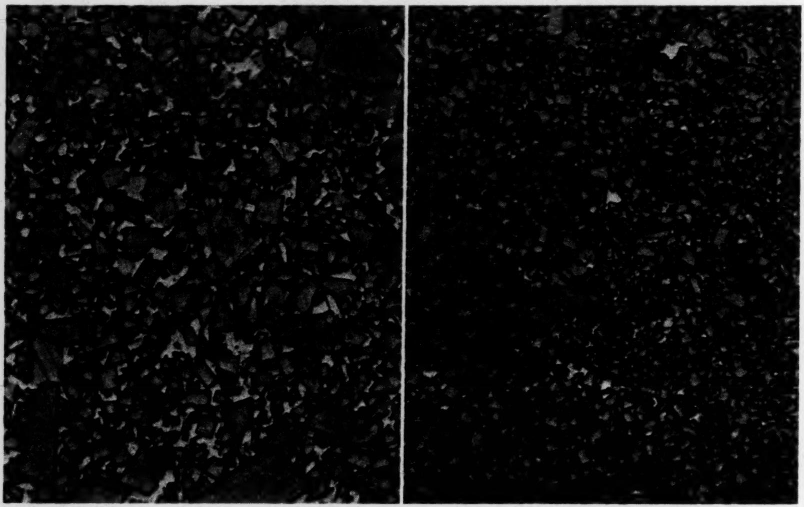


FIG. 8.—Test Block *G*, 88 per cent Tungsten Carbide, 12 per cent Cobalt; Rockwell Hardness A 86.1 Average. Etched in alkaline potassium ferricyanide (×1500).

FIG. 9.—Test Block *J*, 94 per cent Tungsten Carbide, 6 per cent Cobalt; Rockwell Hardness A 92.3 Average. Etched in alkaline potassium ferricyanide (×1500).

Cooperative Test Not Using Same Calibrating Blocks:

Materials.—Four test blocks were manufactured by one cemented carbide producer. The analyses and nominal hardnesses of these blocks are given in Table III. The microstructures of the test blocks are shown in Figs. 8 to 12. Figs. 9 and 10 illustrate microstructural variations of great magnitude present in block *J*.

Preparation of Blocks.—The face areas of the blocks, 1¼-in. square by $\frac{3}{16}$-in. thick, were ground flat and parallel with a diamond wheel. The finish obtained on the testing surface did not exceed 5 microinches rms.

Hardness Testing Procedure.—The procedure for determining the hardness of the blocks was identical with that described previously.

Calibration of Machines.—The hardness machines were calibrated by the internal hardness standard of the company conducting the test. In most cases,

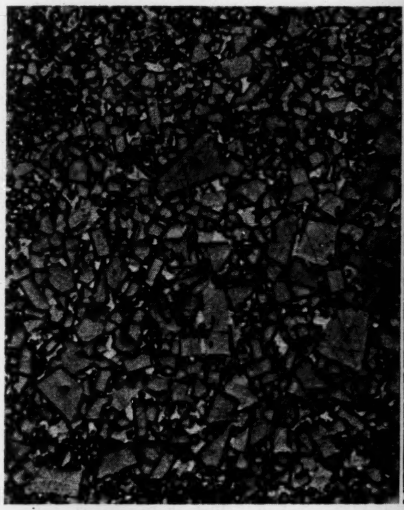


FIG. 10.—Test Block *J*, 94 per cent Tungsten Carbide, 6 per cent Cobalt; Rockwell Hardness A 92.3 Average. Etched in alkaline potassium ferricyanide (×1500).

FIG. 11.—Test Block *H*, 89 per cent Tungsten Carbide, 11 per cent Cobalt; Rockwell Hardness A 89.1 Average. Etched in alkaline potassium ferricyanide (×1500).

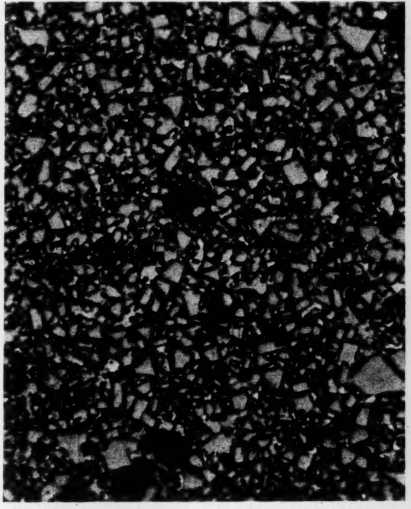


FIG. 12.—Test Block *I*, 94 per cent Tungsten Carbide, 6 per cent Cobalt; Rockwell Hardness A 91.0 Average. Etched in alkaline potassium ferricyanide (×1500).

calibration was conducted at only one hardness level.

Determination of Data.—The hardness of each block was determined in the Quality Control Departments of the participating companies. No special testing method was followed; the company was asked to determine the hardness of the blocks. The companies followed their normal procedure for obtaining the hardness of a random unknown specimen. The high, low, and average hardness values of five readings are given in Table IV.

TEST RESULTS

Statistical Analysis of Data:

The main objective of the statistical analysis of the hardness test data is to establish a range within which the difference between the averages of five tests by any two companies may be expected to fall.

There are three sources of variability in test results. The first of these is the precision of the testing equipment and technique of the operator. The second source of variability is that due to real differences in sample block hardness from point to point and over the area of the sample. In practice, these two effects cannot be separated and the combined effect will be referred to as the precision of measurement. The third source of variability is in the calibration. The methods or standards used for calibration may introduce a real bias for any particular test specimens or the calibration may not be made with sufficient precision.

TABLE IV.—ROCKWELL HARDNESS, A SCALE, VALUES OF CEMENTED CARBIDE BLOCKS (AVERAGE OF FIVE READINGS).

Company	Block *G*			Block *H*			Block *I*			Block *J*		
	Average Reading	Range of Readings		Average Reading	Range of Readings		Average Reading	Range of Readings		Average Reading	Range of Readings	
		Low	High		Low	High		Low	High		Low	High
3.......	86.38	86.0	87.0	89.20	89.0	89.5	91.06	91.0	91.1	92.30	92.2	92.4
7.......	86.1	85.9	86.4	89.1	89.0	89.3	91.2	91.0	91.5	92.5	92.4	92.7
1.......	85.5	85.2	85.9	88.9	88.8	89.1	90.8	90.7	90.9	92.3	92.2	92.3
10.......	86.2	86.0	86.5	89.4	89.3	89.4	91.2	91.0	91.2	92.4	92.3	92.4
8.......	86.3	86.1	86.5	89.3	89.2	89.3	91.0	91.0	91.1	92.4	92.3	92.6
11.......	86.3	86.2	86.5	89.2	89.2	89.2	91.0	91.0	91.1	92.3	92.2	92.4
4.......	85.5	85.3	85.7	88.4	88.3	88.7	90.3	90.2	90.3	91.7	91.7	91.8
5.......	85.8	85.3	86.1	89.1	88.8	89.2	91.2	91.0	91.3	92.3	92.2	92.4
6.......	85.94	85.9	86.0	88.84	88.8	88.9	90.86	90.8	90.9	92.16	92.1	92.2
2.......	87.0	86.8	87.2	89.8	89.7	89.8	91.2	91.2	91.2	92.5	92.3	92.7
9.......	86.1	85.9	86.3	89.2	89.0	89.3	91.0	90.9	91.1	92.2	92.1	92.2
12.......	86.3	86.0	86.4	89.1	89.0	89.2	91.0	91.0	91.0	92.3	92.2	92.4
Block Average..		86.12			89.13			90.99			92.28	

Cooperative Test Using Same Calibrating Blocks:

From these data, an estimate of the precision of measurement at different hardness levels can be estimated by two different statistical techniques, either from the average of the ranges of the five hardness measurements or from an analysis of variance technique. By this latter method, the total variability in the results is divided into three parts: (1) differences between companies (in this experiment these differences are related to the precision of the calibration method), (2) differences in true block hardness in the five areas tested, and (3) a residual error which is testing error and point to point variation in the hardness. If the block is not homogeneous over the entire area, the second method will give a better estimate of the attainable precision for any particular hardness level.

The estimates of the precision of measurement by both methods are given in Table V. In the last column are the 99.7 per cent confidence limits for the average of five tests by any one company. This means that had the company made more than five determinations on the block, the average would be expected to lie in this range.

Control charts of the averages and ranges for the different companies and the limits within which the average of five readings should be are shown in

TABLE V.—PRECISION OF MEASUREMENT, σ.

Block	Rockwell Hardness, A Scale	Average Range	Estimated from Range	From Analysis of Variance	Precision of the Average of 5 Tests[a]
A.....	84.5	0.37	0.159	0.120	±0.2
C.....	85.3	0.28	0.120	0.123	±0.2
D.....	87.1	0.46	0.198	0.134	±0.2
F.....	88.8	0.21	0.090	0.095	±0.1
B.....	91.0	0.17	0.073	0.074	±0.1
E.....	93.1	0.19	0.082	0.093	±0.1

[a] Calculated as $\pm \frac{3\sigma}{\sqrt{5}}$.

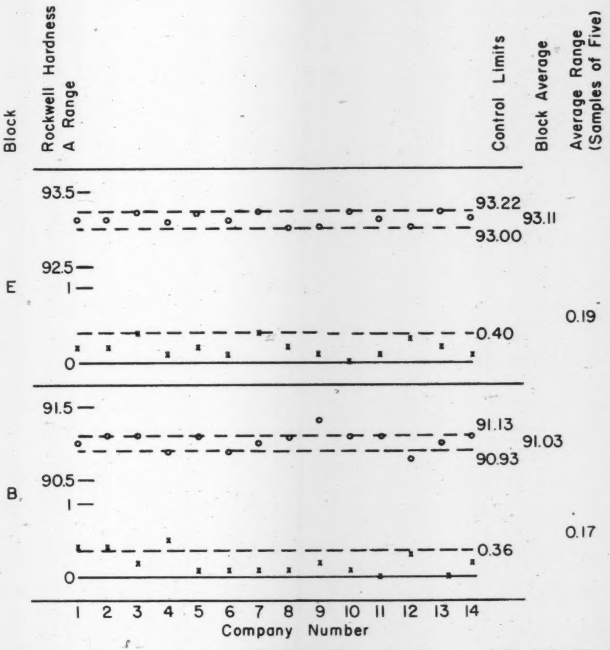


FIG. 13.—Average and Range Charts of Hardness Data for Cemented Carbide Blocks. Rockwell hardness A 91.0 to 93.1.

Figs. 13, 14, and 15. In general, the variability decreases with an increase in hardness as might be expected. This is illustrated in Fig. 16.

The analysis by the method of analysis of variance indicated significant differences among the five areas tested on some of the blocks. In order to explore this apparent inhomogeneity further, test results on the calibration blocks from six of the participating companies were examined. The calibration block corresponding to test block *D* which had such a wide range was not excessively variable, but the average range of tests on the calibration block corresponding to test block *B* was excessively large.

A comparison of the average of hardness ranges for the calibration and test blocks based on results from six companies is shown in Table VI.

Assuming that the inhomogeneities which showed up in this experiment are the exception rather than the rule in practice, confidence limits on the differ-

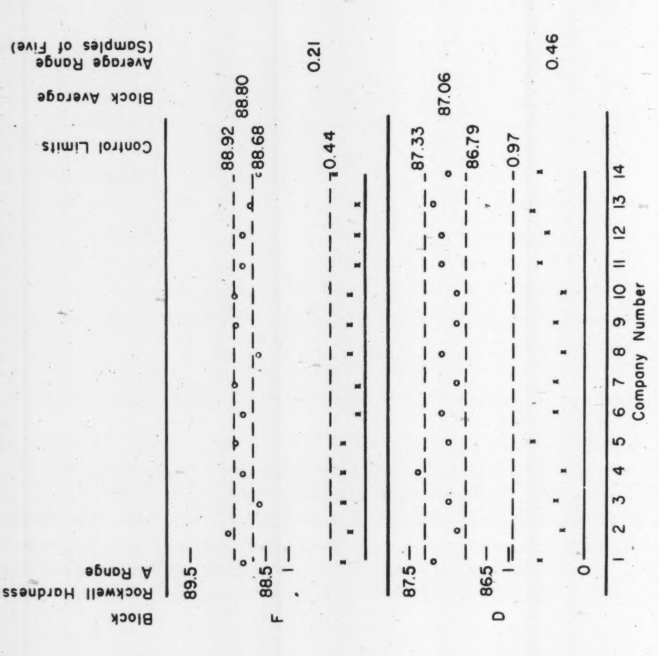


FIG. 14.—Average and Range Charts of Hardness Data for Cemented Carbide Blocks. Rockwell hardness A 87.1 to 88.8.

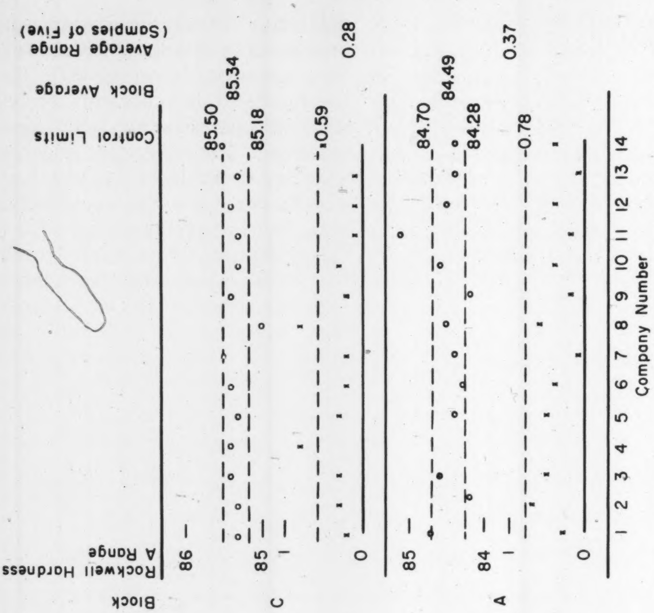


FIG. 15.—Average and Range Charts of Hardness Data for Cemented Carbide Blocks. Rockwell hardness A 84.5 to 85.3.

ence between averages reported by any two companies on a sample can be estimated. This is based on use of the same calibrating method as in the experiment, as those in the first cooperative test and again represent the precision of measurement.

Control charts of these data are shown

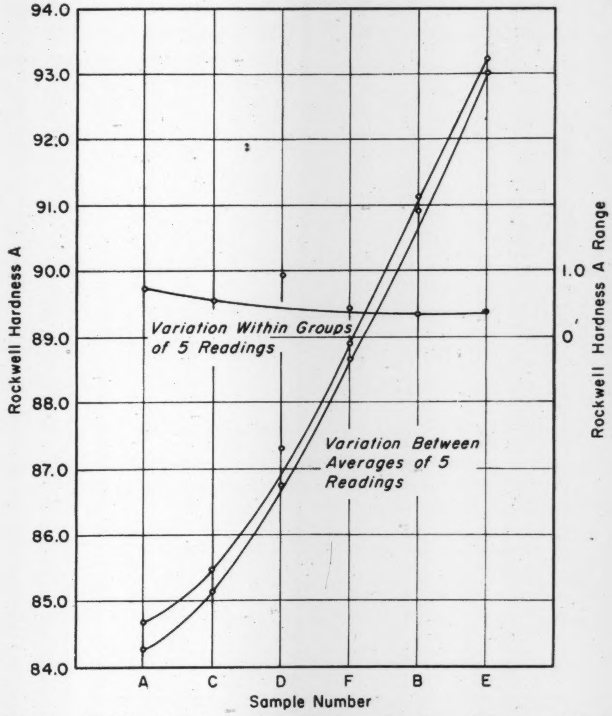


FIG. 16.—Variation Within and Between Five Hardness Readings of Cemented Carbide Blocks.

and 99 per cent of the time the difference may be expected to be less than about 0.6 for the soft material or 0.4 for harder material.

Cooperative Test Not Using Same Calibrating Blocks:

The average hardness and the average of the ranges of five tests by each of the twelve participating companies for the four blocks are listed in Table VII.

The ranges for the different hardness levels are of the same order of magnitude

TABLE VI.—AVERAGE RANGES OF HARDNESS.

Block	Test Block	Calibration Block
*A**	0.3	0.3
*C**	0.2	0.3
*D**	0.5	0.2
F	0.2	0.3
B	0.2	0.6
E	0.2	0.2

* Data from Company No. 4 was omitted in the calculations for these blocks. Their results were significantly more variable than those of the other companies for all three of the softer blocks.

TABLE VII.—HARDNESS VALUES OF CEMENTED CARBIDE BLOCKS.

Block	Average Hardness, Rockwell A Scale	Average Range
G	86.12	0.49
H	89.13	0.23
I	90.99	0.16
J	92.28	0.19

On the basis of these experimental data, the estimate of the difference between averages by any two companies on any sample is up to 1.5 on the soft material and up to 0.8 on hard material.

Since the larger part of the estimated difference is due to differences in calibration, the estimate above, based on tests at only twelve companies, may be unrealistic if these companies are not representative of testing throughout the industry.

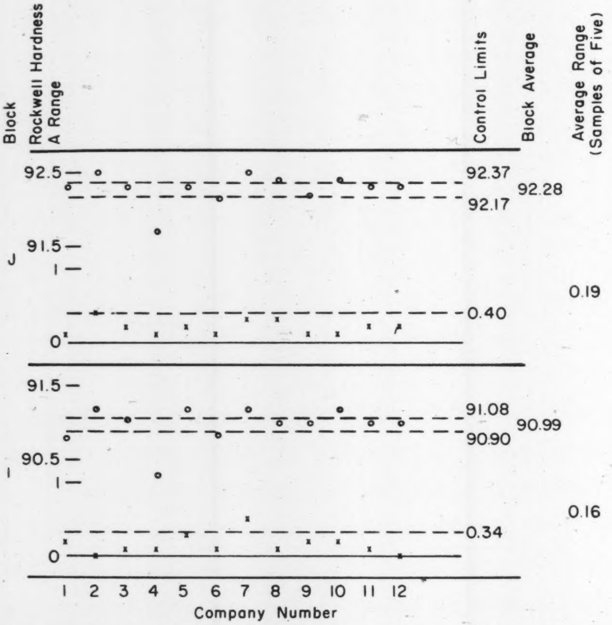


FIG. 17.—Average and Range Charts of Hardness Data for Cemented Carbide Blocks. Rockwell hardness A 91.0 to 92.3.

in Figs. 17 and 18. Comparing these charts with those in Figs. 13, 14, and 15, it can be seen that there is more variability in the averages for the second set of blocks. This can be due to differences in the standards used by each of the companies as well as the heterogeneity of the blocks.

For any company, if the average hardness reported is high or low for any one block, it is generally relatively high or low for the other blocks.

Material Analysis:

It is well known that in the manufacture of cemented carbides great variations in hardness are possible by control of the composition, particle size, distribution, and sintering practice. Accordingly, it is possible to achieve a different hardness level for a given composition by controlling the grain size of the tungsten carbide and this, in turn, may be affected by the sintering temperature, time, and atmosphere.

The blocks used in the testing program were prepared to a given hardness level by various companies using compositions and sintering conditions consistent with their practices. For that reason, a wide variation in composition, particle size, and distribution is evident in the microstructures. For example, test block *A*, shown in Fig. 2, consists of three phases: tungsten carbide, tantalum carbide, and cobalt. With a 27 per cent cobalt content, a Rockwell hardness average of a 84.7 was obtained. If a test block of the same average hardness would be produced by another company, it is possible that a two-phase structure, tungsten carbide and cobalt, with a cobalt content of approximately 30 per cent would be made. With this higher cobalt content, the sintering temperature would be lowered and the possibility of cobalt segregation would be favored. This factor would, of certainty, affect the reproducibility of hardness values obtained in testing the material due to its increased plasticity.

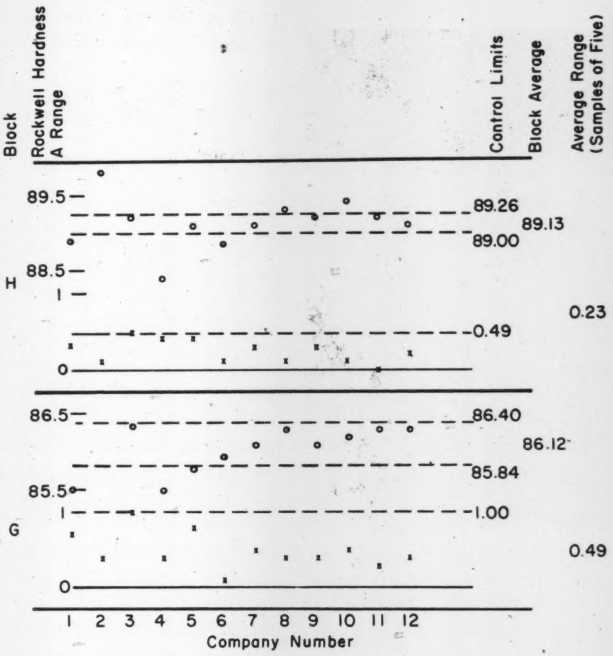


FIG. 18.—Average and Range Charts of Hardness Data for Cemented Carbide Blocks. Rockwell hardness A 86.1 to 89.1.

Further evidence of the effect of microstructure on the hardness level is shown in comparing test block *J* with test block *I*. In both cases, the composition is identical, 94 per cent tungsten carbide and 6 per cent cobalt. However, as shown in Figs. 9 and 12. A difference of 1.2 points on the Rockwell A scale is due to the variation in particle size. Further complications due to porosity evident in Fig. 12 and large particle agglomerates shown in Fig. 11 would affect the range of hardness obtained in either block.

It should be emphasized, however, that the hardness is affected by general rather than isolated microstructural differences. This is indicated by the data given in Table VIII in which the diameters of Rockwell A impressions measured on cemented carbides of different hardnesses are listed.

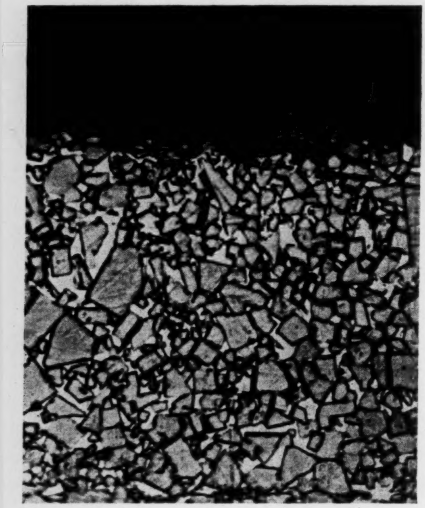


FIG. 19.—Arc of Impression Created by Diamond Brale Penetrator in 87 per cent Tungsten Carbide, 13 per cent Cobalt Cemented Carbide with Rockwell Hardness A 88.0 Nominal Hardness. Etched in alkaline potassium ferricyanide (×1500).

As the carbide particles are generally about 1 to 7 μ in diameter, it is apparent that a great number of particles are beneath the impression. This is further substantiated by the photomicrograph shown in Fig. 19 in which a portion of a Rockwell hardness impression covering a large number of particles is shown..

It is conceivable that variations in microstructure, whether they be due to composition changes, particle size and distribution, segregation of constituents, or porosity, could contribute to the expected range of variability of hardness. It would be desirable, therefore, to minimize this variability in standard test blocks by obtaining as uniform a metallographic structure as possible with a minimum of porosity.

TABLE VIII.—DIAMETERS OF ROCKWELL HARDNESS IMPRESSIONS.

Indicated Hardness, Rockwell A	Diameter, μ
93.2	186
92.5	191
92.2	195
91.0	202
89.5	221
88.8	223
87.8	234

SUMMARY

A cooperative hardness testing program has been undertaken by several cemented carbide producers and one hardness machine manufacturer. Statistical analysis of the results indicates that when common calibrating blocks are used, an expected range of variability between any two companies of 0.4 to 0.6 points on the Rockwell A scale exists. When each company used its own internal hardness test block, the expected range of variability between any two companies was 0.8 to 1.5 hardness points. In both cases, the lower value of the range applied to the hardest test block and the highest value of the range applied to the least hard blocks.

The variables present in the hardness testing of cemented carbides are explained. These include the variables due to the machine, its operation, its operator, and to the metallurgical differences present in the test blocks.

The data presented here, together with the analysis of the data, represent the first phase of a test program designed by the Technical Committee of the Cemented Carbide Producers Assn. The committee is currently investigating the metallurgical variables of test blocks of cemented carbides in the hope that suitable standard test blocks will become available to the industry.

REFERENCES

(1) R. S. Marriner and F. C. P. Mason, "A New Standard Hardness Testing Machine," *Metallurgia*, Vol. 57, No. 399, Jan., 1958, pp. 47–51.
(2) C. A. Bennett and N. L. Franklin, "Statistical Analysis in Chemistry and the Chemical Industry," John Wiley & Sons, Inc., New York, N. Y., (1954).
(3) ASTM Manual on Quality Control of Materials, Am. Soc. Testing Mats. (1951). (Issued as separate publication *ASTM STP No. 15-C.*)

DISCUSSION

MR. LOUIS SMALL[1] (*presented in written form*).—Our company manufactures machines for Rockwell hardness testing and is the largest supplier of diamond penetrators to the carbide industry. We feel that the analysis and procedure described in the paper are excellent but the conclusion, namely, "the expected range of variability of any two companies was 0.8 to 1.5 Rockwell hardness, A scale, points"—is entirely contrary to our experience in the field.

For example, when we sell penetrators to the five carbide manufacturers in the Detroit area, deliveries are made by our field engineer who often uses this occasion to check these penetrators with the customer. The customer will reject those penetrators that are not correct to within 0.2 Rockwell hardness, A scale, points of their internal standards. When the penetrators are outside this limit, the customer's test blocks are at fault or his machine is at fault 95 per cent of the time. We discard the erratic test blocks and we repair the machine at once. The standards of all five companies are practically the same as our master standards, with a difference of less than 0.2 Rockwell hardness, A scale, points on the low block and 0.1 on the high block.

The acceptance of the penetrator is usually made by the man in charge of the department or the most qualified person in the department and not by the people who make the actual routine test. In routine work it is possible for the expected range of variability of any two customers to be between 0.8 to 1.5 Rockwell hardness, A scale, points. However, each carbide company has some man and one accurate machine and at least two or three accurate test blocks and penetrators and can measure the hardness of any piece of uniform carbide within 0.3 Rockwell hardness, A scale, points on the hard grade and 0.6 Rockwell hardness, A scale, points on the soft grade.

We feel that the basic error in these tests was the use of very poor and very erratic blocks for the purpose of standardizing the machines. They were tested in our standardizing laboratory and every single block was found to be entirely too erratic for calibration and testing purposes. Also the number of tests that each company made were not enough for an accurate answer.

---

[1] President, Service Diamond Tool Co., Hardness Testing Department, Ferndale, Mich.

We made additional tests to check this variation by measuring the diameters of the impressions made by the various companies, in block No. 3, square No. 2. In this way we eliminate the relatively large machine error, which is the depth measurement. The loading error is really negligible in comparison. The results of these diameter measurements on the impressions in this square, are in Table III and the confidence limits are $3\ \sigma = 1.44$ optical units. The magnification was chosen so that the last figure of measurement (the 7 of 547) was approximately twice as easy to read as the last figure of the A scale (the 6 of 91.6). This is comparable to our check of block No. 4, section No. 5 which is $3\ \sigma = 0.99$ Rockwell hardness points.

Our analysis of service block No. 1, Rockwell hardness = A92.5 gives $3\ \sigma = 0.462$. This is a poor quality test block that we use for rough preliminary checking. The block is not homogeneous, but by working in very small zones we reduce the errors. This block while twice as accurate as those used in the round robin, is not good enough for the final checking.

We made a further check on two master blocks of a large carbide company and another hardness testing machine manufacturer. The 88.0 block had $3\ \sigma = 0.16$ and 0.18, and the 92.0 block had $3\ \sigma = 0.19$ and 19 for both laboratories. These are two of the very few homogeneous test blocks and are kept in a vault as primary masters against which all secondary masters are checked and evaluated. The deviation for either block is almost ten times less than the average blocks used in the round-robin testing.

We think the report is a great stride forward in the standardization of hardness testing of cemented carbides. We also think that the very erudite statistical analysis may tend to obscure the very bad data obtained. We therefore list general laws of quality control that are applicable.

Whereas in the laboratory one may often be able to hold conditions sufficiently constant that the action of a single law may be observed with high precision, this same degree of constancy cannot in general be maintained under what appear today to be necessary conditions of commercial production. In the majority of cases there are unknown causes of variability which do not belong to a constant system—these so-called assignable causes may be found and eliminated.

Common sense guides us in setting conditions to be satisfied by a cause system in a state of maximum control, which is the condition reached when the chance fluctuations in a phenomenon are produced by a constant system of a large number of chance causes in which no cause produces a predominating effect. Serious difficulties are involved in setting up the necessary conditions for maximum control, so long as one cause does not produce an effect greater than the resultant effect of all the others.

In the general case where X is a chance variable, the effect either of varying the cause Y or of removing this cause must be shown to be significant in the sense of being greater than can reasonably be attributed to the sampling fluctuations in the variable X. The analyst must be successful in choosing the macroscopic cause which is detectable in the objective sense, and must make full use of his powers of imagination, suppositions, idealization, comparison and analogy in the utilization of all available data.

In any program of control we must start with observed data—good, bad, or indifferent. We are frequently confronted with expressing results obtained by empirical methods in the hands of fallible operators on more or less representative samples of generally very heterogeneous materials. The precision then is based on three factors which have not been controlled—the authenticity of the sample, the operator, and the method itself. The usefulness of refined methods of analysis is very questionable when the data are not good. And good data in general are expensive. In the process of getting them many measurements are usually taken, from which a few are finally chosen as being good.

MR. J. MANDEL[2] (*presented in written form*).—To anyone interested in the design and analysis of interlaboratory studies, a paper dealing with this subject and containing extensive data is always an object of eager attention. The paper presented is a serious study of a test method. Several of its aspects are of general interest.

Referring to the first part, six sets of duplicate blocks were tested by 14 laboratories. The authors refer to one of each set of duplicates as the calibration block and to the other as the test block. An analysis is made to determine the variability of the results on the test blocks, after correcting them on the basis of the supposedly identical calibration block. Such a calibration procedure is, of course, somewhat unusual. A more common method is to use a calibration curve obtained on an entirely different set of materials. The difference is not trivial. In order to see this, I refer you to the literature in chemical analysis. Many papers dealing with the study of spectrophotometric methods of analysis contain a section on precision and accuracy. Now, most of these methods of analysis require a calibration curve. A measurement is made and the result is then read from the calibration curve using the measurement as one of the coordinates of a point on the curve. Why should such a result lack accuracy? Of course, it can lack precision because of random errors. But it seems, superficially at least, that it cannot be inaccurate, that is, be subject to systematic bias, since the calibration curve defines the true value. Why then, is there a need for the study of accuracy in such cases? The answer is that the calibration curve in question is actually only one of an entire set of calibration curves. Indeed, factors other than the concentration of the solution may affect the results. Among those factors, interfering substances are of special interest. If now the calibration curve is obtained for samples containing one type of interfering substance, and the sample to be analyzed contains another type of interfering substance, a bias may very well result.

On the other hand, if the samples used for calibration and for testing are identical, as they were in the present study, one would expect the results of all laboratories to agree to within the experimental error within laboratories. Thus, by calibrating in this manner one essentially washes out all laboratory differences. The control charts for the first part of the study show this clearly.

One could go beyond the author's analyses by analyzing the uncorrected values of the test blocks, or, alternatively, the original observations on the calibration blocks. Such an analysis would reveal the real laboratory-to-laboratory differences.

What does the second part of the paper contribute? We have just seen that the first part also gives information on laboratory-to-laboratory variability. It is not entirely clear to me whether in the first part, the work of each laboratory was supposed to be under more rigid control than in the second part. If so, the comparison of the two parts, making no use of calibration corrections, is of real interest. Unfortunately, the paper does not contain such a comparison.

There is one final point I wish to raise. The authors observe that the range of the five readings on a block decreases as the average value for the block increases. In such cases, that is, whenever the standard deviation of the replication error is a function of the magnitude of the measurement, an analysis based on a transformed variable is easier to interpret and generally more informative than without transformation. I rather suspect that in the present case too, such an analysis would be of interest.

---

[2] Testing and Specification Section, Division of Organic and Fibrous Materials, National Bureau of Standards, Washington, D. C.

MR. B. F. SHEPHERD.[3]—How many tests can be made upon a Brale without using a correction factor or replacement, that is, what is the life of a Brale on tungsten carbide?

MR. O. W. REEN (*author*).—I believe either Mr. Small or Mr. Lysaught of Wilson Mechanical Instrument Co. would be much better able to answer that question than I could.

MR. RALPH G. KENNEDY.[4]—I would like to add the same comment as the last discusser. It would like to see the data of Table II expressed as were the data for the information in Table IV, that is, where the calibrations were made against the company's own internal standards and not against the somewhat faulty calibration blocks that were furnished in this series.

MR. O. W. REEN (*author*).—We were a little dismayed when it was determined that we did not have good calibrating blocks. Yet, if we examine the ranges reported from the various companies for each test block, they were not too bad. It was when we compared the average hardness values reported by all of the companies that a 0.4 to 0.6 Rockwell A hardness variation resulted. The ranges reported by the individual companies were within 0.4 Rockwell A points as shown in Table II.

As far as the mathematics are concerned, I would like one of my coauthors —Miss Caugherty—to answer Mr. Mandel's discussion.

MISS BETTY CAUGHERTY (*author*).—The answer to the one point that Mr. Mandel raised—what would have happened had we not used the calibration block. Correction—on the first 6 blocks as compared with the last 4 blocks. We had calculated that but it was not presented in the paper. Actually the results are quite similar. The first set of blocks were carefully tested and readings made under good control—the second set just a random set of blocks sent in to a quality control testing department. In the range that you might expect on any two companies on the first 6 blocks, without the use of the special calibrating blocks was from about 0.8 for the harder blocks to about 1.2 for the softer ones. This agrees pretty well with the range we found for the four random blocks.

The second question which the discusser raises about setting up the calibration curve—as I understand it, in hardness testing this is not a general practice, so I do not know whether there would be any advantage to looking into this or not.

MR. VINCENT E. LYSAGHT.[5]—Experience shows than an average figure would be about 1000 to 2000 tests per diamond, that is, using the Rockwell hardness A scale and not the heavier load (C scale) which is sometimes used. In connection with this paper, I would like to say that the study has been so carefully planned and executed it is difficult to criticize or offer suggestions, but I think we ought to keep in mind the dimensions of the values that we are talking about. The measurements of 0.4 to 0.6 when the machines were used with calibrated blocks, whether they are the correct standards or incorrect, but nevertheless were all the same, is only in the neighborhood of 0.00004 in depth of indentation. This is a very small depth as we know and I think that the work that this group had done has turned out remarkably well when dealing with such small measurements.

---

[3] Metallurgist, Ingersoll Rand Co., Phillipsburg, N. J.

[4] Director of Laboratories, The Cleveland Twist Drill Co., Cleveland, Ohio.

[5] Sales Manager, Wilson Mechanical Instrument Division, American Chain and Cable Co., Inc., New York, N. Y.

MISS BETTY M. CAUGHERTY AND MESSRS. H. T. OATMAN AND O. W. REEN (*authors' closure*).—We were fortunate in having Mr. Small's laboratory and services available for conducting some rather exhaustive tests on the calibration blocks we prepared. His findings substantiated, for the main part, our results that the blocks should be more uniform.

As to the acceptance of the penetrator by a customer, it was stated that a qualified technician should measure the hardness of any piece of uniform carbide within 0.3 Rockwell hardness, A Scale points on the hard grade and 0.6 Rockwell hardness, A Scale on the soft grades. An examination of the ranges obtained by each individual company shown in Table II shows that, for the most cases, the hardnesses fell within the stated limits. The fact that great variability existed between results of different companies indicates variability other than the test blocks. In regard to the testing personnel, each block was tested by the technician doing the majority of testing for his company.

Pertaining to the quality of the test blocks, we feel that blocks A through E represent normal microstructures with the usual amount of porosity. Blocks G through I were not designed to be used as test blocks. Although the blocks can be of excellent metallurgical quality, it is entirely possible that they are of poor test block quality. As cemented carbides are not homogeneous in structure, consisting of two, three or more phases, the problem of obtaining uniform hardnesses over the entire testing surface is difficult. This problem is further magnified by the fact that different producers create grades of the same hardness by varying the particle size of the carbide and the cobalt content. All of these grades are found in industrial use, and it is desirable to know their hardnesses.

Of course, it would be most desirable to have a standard test block with the least hardness variation over the entire test surface. We are currently in the process of evaluating an entirely new set of test blocks. The testing of these blocks has been statistically planned to determine the uniformity of the material and the effect of surface finish. If these blocks prove to be homogeneous, it is our aim to provide these to the industry as primary standard test blocks.

One question which Mr. Mandel asks is the comparison between the first set of six blocks making no use of the calibration corrections and the second set of four blocks. These calculations were made, though they do not appear in the paper. The first set of blocks were tested under carefully controlled conditions, whereas the second set were submitted at random and tested as routine samples in each company's Quality Control Testing Department. Actually, the results on the two sets were in good agreement. The range that might be expected between any two companies based on the first without the calibration corrections is from about 0.8 points of Rockwell hardness A for the harder blocks to about 1.2 points of Rockwell hardness A for the softer blocks.

The second question concerns the method of correction used. It is our understanding that calibration curves such as are common in chemical analysis procedures are not the general practice in hardness testing. Unlike chemical systems where an independent standard can be devised (such as solutions of varying but known ion content each of which are reasonably homogeneous), we have no independent homogeneous reference standards at these hardness levels. Consequently, it is not feasible to develop calibration curves.

EXPERIMENTAL OBSERVATIONS ON THE RELATION BETWEEN POLARIZATION RESISTANCE AND CORROSION RATE*

By Milton Stern[1] and Edward D. Weisert[1]

Synopsis

A method for determining corrosion rate from electrochemical measurements is described. The method involves use of a parameter called polarization resistance, which is the slope of the polarization curve in the region very close to the corrosion potential. It is shown that corrosion rate is inversely related to polarization resistance. For most corroding systems, the corrosion rate can be estimated to within a factor of 2 by a simple measurement of the current required to polarize a few millivolts. Some knowledge of the electrochemistry of the system permits a much better estimate. An experimental calibration may be required in some cases. However, the calibration may be no better than the method of estimating described here, since measurement of instantaneous corrosion rate is particularly difficult. Data from a variety of published sources, along with a number of new observations, are used to support the correlation. The data presented extend over six orders of magnitude.

A rapid and convenient method for estimating changes in corrosion rate without disturbing the system by the measuring method is highly desirable. The corrosion rate of a metal may change continuously with time because of several factors. Among these may be included changes in the nature of insoluble corrosion products which may accumulate on the surface; changes in composition of the environment because of the corrosion process; or changes in the real area of the metal surface due to such effects as preferred grain boundary attack. Other more subtle phenomena may also cause changes in corrosion rate with time. Unfortunately, obtaining the weight loss of a metal over a given increment of time does not yield a true corrosion rate but rather produces an average rate over the time interval selected. Also, intermittently removing the sample for weight change measurements often affects the subsequent corrosion rate upon re-exposure to the environment.

Chemical analysis of the solution also presents difficulties, particularly when the rate changes markedly with time or when corrosion products are insoluble. Also, this method is tedious and may not be sufficiently sensitive to detect changes during the early stages of corrosion.

Determination of corrosion rate by

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Metal Research Laboratories, Union Carbide Metals Co., Division of Union Carbide Corp., Niagara Falls, N. Y.

measuring the increase in electrical resistance of a wire or ribbon-type sample due to a decrease in dimension is not always possible. The method requires extremely accurate temperature control or a comparison sample which is completely protected from the environment. Also, materials studied must be capable of fabrication into the desired special shape. This method is only quantitative if the corrosion process produces very uniform attack.

Thus, it is evident that present methods are not always adequate. Another procedure which will produce continuous measurement is desirable.

DESCRIPTION OF METHOD

In a study of the corrosion of steel and cast iron in natural waters, Skold and Larson **(1)**[2] observed that a plot of corrosion rate *versus* "polarization resistance"[3] on logarithmic scales gave a straight-line relation. Corrosion rate increased as "polarization resistance" decreased. Their conditions were such as to give corrosion rates which varied over several orders of magnitude.

Stern **(2)** showed that the method has foundation in theory and also provided some supporting evidence. Since the procedures have been described in detail, they will only be summarized here.

For a system controlled by activation polarization,[4] the following relation applies:

$$\frac{\Delta E}{\Delta I} = \frac{\beta_a \beta_c}{(2.3)(I_{\text{corr}})(\beta_a + \beta_c)} \quad \ldots\ldots (1)$$

where $\Delta E/\Delta I$ is the polarization resistance, β_a and β_c are the slopes of the logarithmic *local* anodic and cathodic polarization curves, and I_{corr} is the corrosion current.

It is important to note that this equation only applies when ΔE is very small. For most cases, the change in potential, ΔE, from the corrosion potential should be less than 10 mv. If the metal is polarized more than this, the relation between potential and applied current will not necessarily be linear, and a proper value of the slope, $\Delta E/\Delta I$, will not be obtained. The extent of the linear region for various systems has already been analyzed **(2)**.

For a system whose corrosion rate is controlled by concentration polarization of the reduction reaction, Eq 1 reduces to

$$\frac{\Delta E}{\Delta I} = \frac{\beta_a}{2.3 I_{\text{corr}}} \quad \ldots\ldots\ldots\ldots (2)$$

Note that if one considers the β values constant, both Eqs 1 and 2 show that polarization resistance is inversely proportional to corrosion current. Thus, if the polarization resistance doubles, the corrosion current is reduced to half its value. Very often one works with systems where corrosion rates vary over several orders of magnitude. Thus, plotting a calibration curve on linear paper crowds much of the data. Therefore, it is most convenient to plot the data on logarithmic scales as was done by Skold and Larson **(1)**. Taking the logarithm of both sides of Eq 1 yields:

$$\log \frac{\Delta E}{\Delta I} = \log \frac{\beta_a \beta_c}{2.3(\beta_a + \beta_c)} - \log I_{\text{corr}} \quad \ldots (3)$$

Thus, a plot of polarization resistance *versus* corrosion current on logarithmic scales should yield a straight line with a slope of −1. If one assigns values to the β constants, a plot of Eq 3 may be constructed. Figure 1 shows such a relation assuming both β constants equal 0.1 v. This plot represents a calibration for such an ideal system. Measurement of polarization resistance permits a direct determination of corrosion current. However, to use this method, the β values must be known and they must remain constant.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] Polarization resistance is the slope of the linear portion of a polarization curve obtained in the potential region very close to the corrosion potential.

[4] For systems where the local anodic and cathodic polarization curves are logarithmic in nature; that is, where the rates of the oxidation and reduction reactions involved in the corrosion process are logarithmic functions of potential.

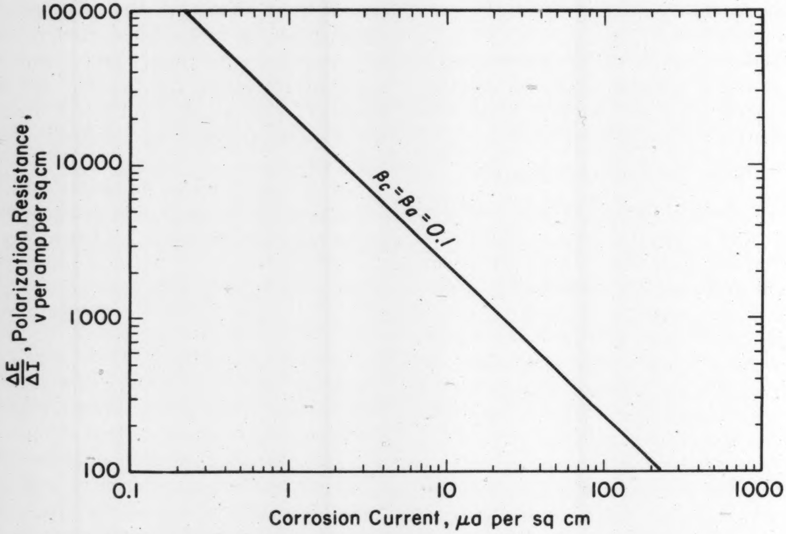


FIG. 1.—Calculated Relation Between Polarization Resistance and Corrosion Current for a System with Equal Cathodic and Anodic β Values of 0.1 v.

ERRORS IN METHOD WHEN VALUES OF BETA CONSTANTS ARE UNKNOWN

Assume that the β values remain constant for a given system but that their exact values are not known. A study of available data on activation polarization kinetics reveals (3)[5] that the range of experimental values of β is limited. This applies to a large variety of electrochemical reactions. In general, β values range between 0.03 and 0.18 v. Actually, values of 0.03 v are quite rare and generally apply to hydrogen overvoltage on materials like platinum and palladium. More practical engineering materials exhibit larger values of β for this reaction. A β value of 0.18 v is also rare for activation overvoltage. It is safe to say that the large majority of β values lie between 0.06 and 0.12 v. It is important to note from Eq 1 that doubling one of the β values increases the proportionality constant between polarization resistance and corrosion current. However, the proportionality constant is not doubled because each β value appears in both numerator and denominator. Thus, while the relation between polarization resistance and corrosion current is sensitive to change in either β value, it changes less than a given change in either β value. For example, consider a system with a β_a value which changes from 0.06 to 0.12 v and a constant β_c of 0.06 v. All

---

[5] This reference gives data which permit calculation of β values for a variety of processes on many metal surfaces.

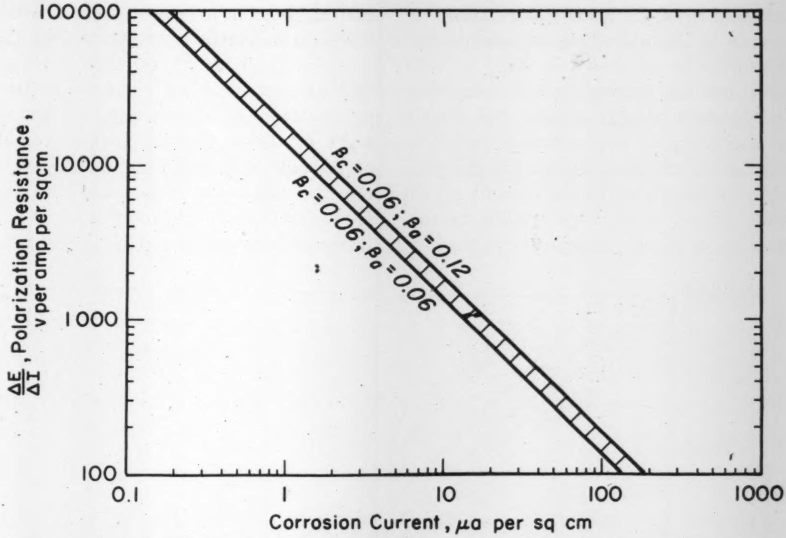


FIG. 2.—Limits Within Which the Relation Between Polarization Resistance and Corrosion Current Applies for a System with a Constant Cathodic β Value of 0.06 v and an Anodic β Value Which Varies from 0.06 to 0.12 v.

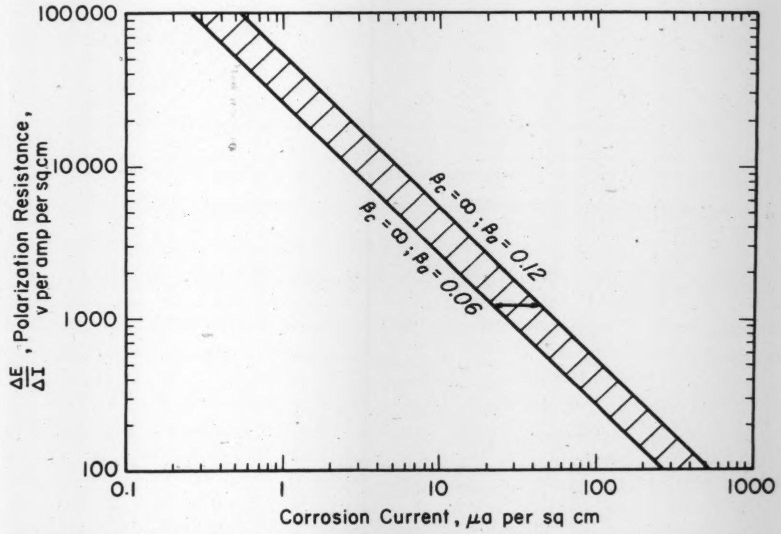


FIG. 3.—Limits Within Which the Relation Between Polarization Resistance and Corrosion Current Applies for a System with a Constant Cathodic β Value of Infinity (a limiting diffusion current) and an Anodic β Value Which Varies from 0.06 to 0.12 v.

the points which relate polarization resistance to corrosion current should fall within the band shown in Fig. 2. Note that if one has reasons for believing that the β values of this system fall within the above limits, then a simple measurement of polarization resistance will provide a calculated corrosion current within about 20 per cent. This applies to corrosion rates of any magnitude regardless of whether they are so small that they cannot be detected by conventional methods or so large that the sample disappears in a few hours.

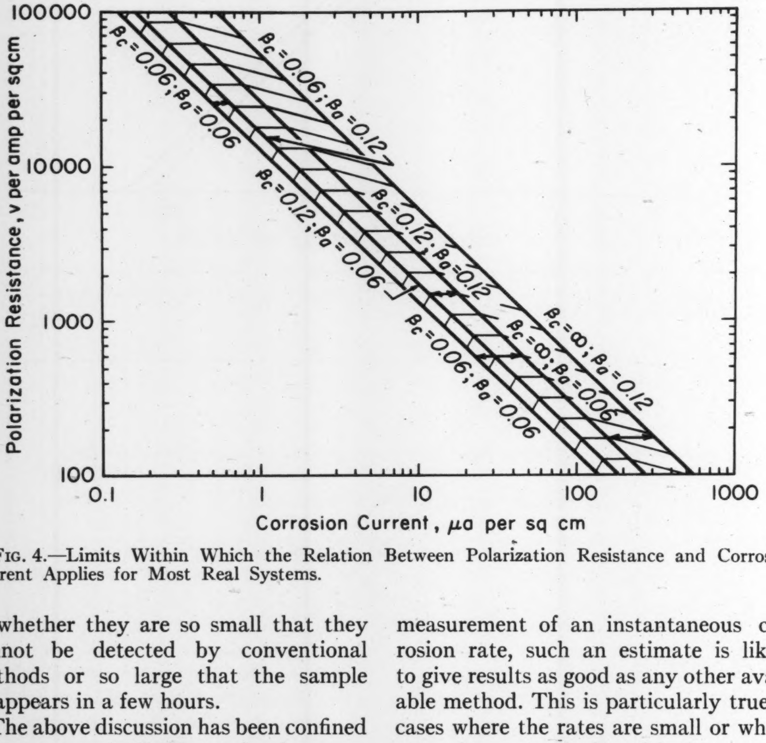


FIG. 4.—Limits Within Which the Relation Between Polarization Resistance and Corrosion Current Applies for Most Real Systems.

The above discussion has been confined to systems controlled by activation overvoltage. Many systems, however, are controlled by a cathodic limiting diffusion current. In this instance, the slope of the cathodic polarization curve, β_c, may approach infinity. This is the situation where Eq 2 applies. Thus, one may consider a system where β_c equals infinity and β_a varies from 0.06 to 0.12. The band for such a system is shown in Fig. 3. It is wider than that illustrated in Fig. 2, and measurement of a given value of polarization resistance permits an estimate of corrosion rate within a factor of 35 per cent. Such an estimate may not be adequate for some purposes. However, when one considers the extreme difficulty entailed in experimental measurement of an instantaneous corrosion rate, such an estimate is likely to give results as good as any other available method. This is particularly true in cases where the rates are small or where rates change markedly with time.

Figure 4 shows a variety of bands, the total width of which describes all systems where β_c varies from 0.06 to infinity and β_a varies from 0.06 to 0.12. Inspection of Eq 1 shows that it also includes systems where β_a varies from 0.06 to infinity and β_c varies from 0.06 to 0.12. A large majority of real systems fall within this over-all band. Thus, without any information at all concerning the β values of a given system, the corrosion rate can only be determined within a factor of two. However, with some knowledge of the electrochemistry of the system, which permits an estimate of β values, the band can be narrowed considerably as described above.

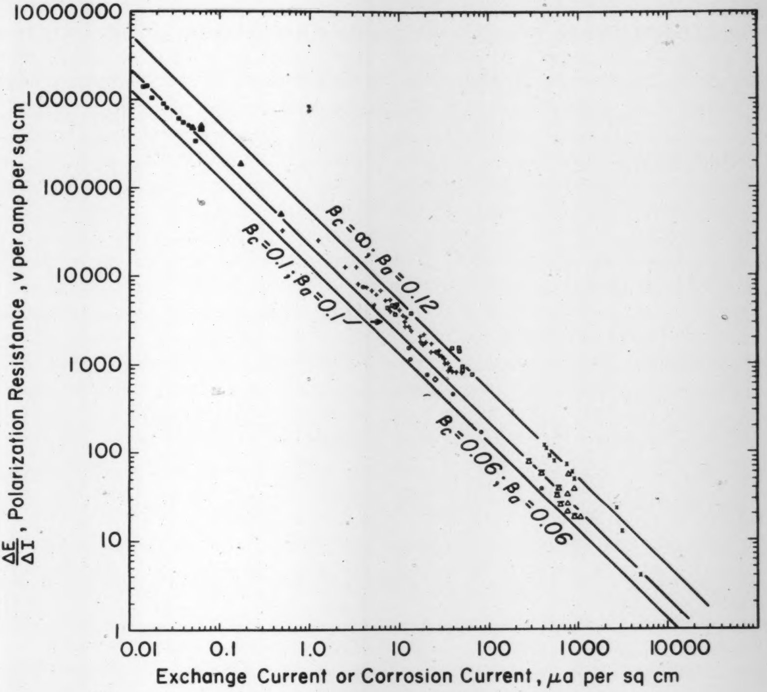


FIG. 5.—Experimental Observations.

Open circles and triangles represent nickel in hydrochloric acid (**4,5**); open diamond represents iron in dilute hydrochloric acid containing sodium chloride (**8**); x's represent various steels in sulfuric acid (**9**); open squares represent steel and cast iron in high conductivity waters (**1**); crosses represent cast iron in natural and synthetic waters (**10**); solid circles (**11**), solid triangles (**12**), and solid squares (**13**) apply to ferric-ferrous exchange current on a variety of passive surfaces.

EXPERIMENTAL OBSERVATIONS

A variety of experimental observations are available to test the general validity of this method of estimating instantaneous corrosion rates. Actually, some of the data which can be used come from reversible electrode systems. These systems are not corroding but rather have an exchange current, I_0, which for these purposes can be considered similar to a corrosion current; that is, for a reversible system, the corrosion current in Eqs 1 or 2 is replaced by the exchange current.

Data from six different published sources along with four sets of unpublished data are shown in Fig. 5. The solid points apply to exchange currents, while the open points are corrosion currents. Note that the data range over six orders of magnitude. The extremities of the band shown in Fig. 4 are included along with a calculated line assuming equal anodic and cathodic β values of 0.1 v.

The points indicated by open circles are unpublished data of Young (4) which were obtained for nickel in nitrogen-saturated hydrochloric acid. Concentration varied from 1 to 10 per cent HCl at 50 C. This system is controlled by activation polarization.

The points indicated by open triangles are unpublished data of Geary (5) and apply to nickel in hydrogen-saturated 3.2 *M* hydrochloric acid at 50 C. The points differ in that pure nickel from a variety of sources was used. It was also found that traces of impurities in the solution affected corrosion rate and electrochemical properties. In these experiments, small quantities of solution were periodically removed for chemical analysis to determine corrosion rate. At the end of the test, anodic and cathodic polarization data were obtained which gave both polarization resistance and β_c and β_a. Cathodic β values ranged from 0.10 to 0.12. Anodic β values ranged from 0.07 to 0.09 with most close to 0.08 v.

Open triangles with a horizontal bar are also Geary's data for the same system as described above. However, in this instance, corrosion current was determined by another electrochemical method (6,7). This involves extrapolation of the cathodic Tafel overvoltage curve to the corrosion potential. In general, corrosion rates determined by this method compare more favorably with the expected position on Fig. 5 (calculated from a knowledge of the β values) than do results of the analytical data. This probably results from several effects. First, corrosion currents determined by this extrapolation are obtained at essentially the same time as the measurement of polarization resistance; that is, polarization resistance and cathodic overvoltage data are obtained from different parts of the same experimental polarization curve. Also, the analytical data were obtained over a period of at least 24 hr, and then the electrochemical data were obtained. Thus, there is some difference in the time at which the data are obtained. It is important to appreciate that in the analytical method one usually obtains a number of experimental points of weight change as a function of time. Often there is sufficient scatter in the data to mask real changes in rate, and one is often only justified in placing a straight line through the points. This, then, assumes the rate is constant with time when, in fact, changes may occur. This phenomenon was observed in the experiments of Young (4), who followed polarization resistance continuously and simultaneously periodically removed solution for analyses. Smooth, continuous changes in polarization resistance were observed, yet the chemical analysis data showed sufficient scatter to justify only a constant corrosion rate. Young's points on Fig. 5 represent this measured corrosion rate plotted against the average polarization resistance obtained over the time interval of the experiment.

Other points on Fig. 5 represent iron in NaCl at pH 1.5 and 25 C (8) and various steels of different composition in sulfuric acid (9). These latter data by Bonhoeffer and Jena are controlled by activation polarization and yet they fall on the high side of the band in Fig. 5 where concentration polarization is indicated. The reason for this is not known.

The data of Skold and Larson (1) for corrosion of steel and cast iron in high conductivity waters are indicated as open squares. The reaction here is most likely controlled by oxygen diffusion, and the points lie in the proper region of the band.

Extensive tests by Stumm (10) with

cast iron in three synthetic and two natural waters provide a large number of points (indicated as crosses). The work was done in such a manner as to eliminate an IR drop in the polarization measurement. These data, which represent over 50 experimental points, are described by Stumm in an equation of the form $\frac{\Delta E}{\Delta I} = \frac{0.034}{I_{\text{corr}}}$. This system is quite consistent with the theoretical treatment described above. The equation on a logarithmic basis has a slope of -1. In addition, the constant of 0.034 v is very close to what one might predict if he did not have this experimental calibration available. This constant represents $\frac{\beta_a}{2.3}$ in Eq 2, since the system is undoubtedly controlled by an oxygen limiting diffusion current. Stern and Roth (8) have reported values of β_a for iron of 0.068 and 0.078 v measured by two different techniques, while Kaesche and Hackerman (7) report a value of 0.075 ± 0.01 v. Considering the value is 0.075 v, the calculated constant in Stumm's equation above is 0.033—in unusually good agreement with his value of 0.034. Examination of these data plotted in Fig. 5 reveals that they lie in that portion of the band which is required of a system with a β_c value of infinity and a β_a value of about 0.075 v.

The remaining solid points on Fig. 5 all apply to ferric-ferrous exchange currents on a variety of passive surfaces. Detailed descriptions of the particular systems are available (11,12,13). In general, the β values vary from 0.09 to 0.12, but a few systems exhibit values as high as 0.15.

Inspection of Fig. 5 reveals remarkably good agreement between experimental observation and the predicted behavior described by Eq 1, particularly when one considers that the experimental data range over values which differ by 10^6.

It is worth noting that the particular advantage of the log-log plot used here is that deviations from a line of a given distance represent deviations of a constant per cent of the total value of the variable at that point, irrespective of the part of the plot in which they lie. In contrast to this, deviations of a given amount from a plot with ordinary coordinate paper represent a constant numerical deviation, which may be a large per cent when the variable is small and a small per cent when the value of the variable is large. Thus, a logarithmic plot is of particular assistance in representing the data of Fig. 5, since it presents the data with the same percentages of accuracy, no matter what the magnitude of the quantities may be.

DISCUSSION

Successful use of the method described here requires a thorough knowledge of the basic principle. It is necessary to measure polarization resistance very close to the corrosion potential. Preferably, the maximum amount of polarization should be 10 mv and ideally, if proper instrumentation is available, 5 mv should not be exceeded. It is important to realize that the amount of current used to polarize is not the determining factor in obtaining a good value of polarization resistance, but rather Eqs 1 and 2 are derived for conditions where the potential change is small. A convenient procedure for rapidly measuring polarization resistance simply involves determining that current required to polarize 5 mv. This permits calculation of polarization resistance and can be done at any desired time interval. Unlike another electrochemical method for measurement of corrosion rate (6,7), this procedure is not likely to affect the subsequent corrosion rate of the material by changing the nature of the surface due to marked potential changes from the corrosion potential. This is a particularly important advantage.

Measurement over a very small potential increment has further advantages. The linear relation between potential and applied current, very close to the corrosion potential, exists because the difference between two logarithmic functions of current approximates a linear function when the logarithmic functions are of the same order of magnitude. That is, the applied cathodic current is the difference between the total cathodic and total anodic currents at any potential:

$$I_{\text{applied}} = I_c - I_a \qquad (4)$$

If I_c and I_a are logarithmic functions of potential, their difference approximates a linear function of potential when they are of similar value. However, in some real systems, I_c or I_a, may not be a truly logarithmic function of potential (for example, in the potential region where both activation and concentration polarization are important). Therefore, a linear dependence of potential on applied current might not be expected. Nevertheless, when one considers only a 5-mv range for such a situation, the local polarization curves will often sufficiently approximate logarithmic behavior to produce the desired linear behavior. Therefore, one need not have exactly logarithmic local polarization curves to obtain useful results. However, under such circumstances, the slopes of the logarithmic curves, β_c or β_a, need not have the values described above for activation polarization; rather, they could be much higher. Thus, such conditions would be expected to produce results which fall near the top of the band shown in Fig. 5.

Two difficulties are associated with use of this method without proper knowledge of the β values. One involves errors due to a resistance drop in the measured polarization value. Such an effect would produce calculated polarization resistances which are too large. This problem was recognized by Skold and Larson in their work in natural waters. It would produce a calibration curve which does not have a slope of -1 as illustrated here. Rather, the slope would be less than this.

Perhaps a more serious difficulty is associated with one of the basic assumptions made above. Systems considered so far have been those where, because of some environmental or metallurgical change, the corrosion rate changes. However, a given set of β values has been assumed to be constant during the change. This is equivalent to considering that the local polarization curves shift to the left or right (or up and down) without any change in slope. This is undoubtedly not the case for some real systems. For example, consider iron corroding in a solution where the rate is controlled by the limiting diffusion current for oxygen. If one adds increments of a corrosion inhibitor which reduces the corrosion current gradually below the limiting diffusion current of oxygen, then the cathodic β value would be expected to decrease from a high value to a normal activation overvoltage value. This would produce a relation between polarization resistance and overvoltage which is not as simple as that shown in Fig. 1. The line need not be straight and, at any rate, would have a slope less than -1. This can be seen by inspection of Fig. 4. For high corrosion currents, the points would be expected to fall in that part of the band where β_c is near infinity. For low corrosion rates, the relation should fall solely in the activation overvoltage range of the band. This factor may have been important in Skold and Larson's (1) system. Recently, Kobayashi and Nagayama (14) reported on the relation between polarization resistance and corrosion rate. They worked with mild steel in 1 per cent sodium chloride containing various quantities of sodium silicate. The effect of adding certain metal chlorides to the system was also investigated. They report a straight-line relation between polarization resistance and corrosion current. However, their slope is not theoretical, but rather, similar to that of Skold and Larson. The factors described above may have caused this.

Perhaps another factor which might cause difficulties in the application of this method should be mentioned. The polarization resistance is really inversely related to the current at the intersection of the total anodic and cathodic polarization curves. In most cases, this is the corrosion current; however, in some instances, another oxidation process may occur in addition to the metal dissolution reaction. Thus, the calculated current is really equivalent to the total rate of oxidation which will be greater than the rate of metal corrosion. Some knowledge of the chemistry of the system should eliminate difficulty here.

The following is a list of situations where it appears that the use of linear polarization measurements can supply valuable information:

1. Studies of the effect of environment variables on corrosion rate. These include changes in composition, velocity, and temperatures.
2. Evaluation of inhibitors in controlling corrosion.
3. Comparison of the corrosion rates of various alloys of similar composition in a given environment.
4. Determination of changes in corrosion rate with time, including studies of underground structures as well as materials in aqueous solutions.
5. It also may be possible to evaluate the condition of coatings in service which cannot be inspected by visual methods.

While the use of polarization resistance to determine corrosion rates cannot be considered a universal approach, there is sufficient basis in theory along with supporting evidence to believe that the technique will find a particularly useful place in corrosion measurements.[6]

SUMMARY AND CONCLUSIONS

Corrosion rate is inversely related to polarization resistance. For most corroding systems, the corrosion rate can be estimated to within a factor of 2 by a simple measurement of the current required to polarize a few millivolts. Some knowledge of the electrochemistry of the system permits a much better estimate. An experimental calibration may be required. However, the calibration is no better than the experimental method used to determine instantaneous corrosion rate. For many systems, this is particularly difficult and the results may not be better than the method of estimating described here.

Calibration is required if an *IR* drop is included in the measurement. An *IR* drop may result from either use of a low-conductivity electrolyte or from high resistance films on the metal surface. The method cannot be used in those cases where an oxidation process occurs which is different from the metal dissolution reaction.

Acknowledgment:

The authors acknowledge the cooperation of S. G. Young and A. L. Geary, who provided experimental data for inclusion in this work. Werner Stumm kindly provided permission to quote his yet unpublished data.

---

[6] Since this manuscript was written, a method for measuring corrosion rates described by Engell in aerated solutions has appeared (**15**). It applies only to conditions where the cathodic process is controlled by a limiting diffusion current and is essentially a special case of the method described here where β_c approaches infinity.

REFERENCES

(1) R. V. Skold and T. E. Larson, "Measurement of the Instantaneous Corrosion Rate by Means of Polarization Data," *Corrosion*, Vol. 13, p. 139 (1957).

(2) M. Stern, "A Method for Determining Corrosion Rates from Linear Polarization Data," *Corrosion*, Vol. 14, p. 440t (1958).

(3) J. O'M. Bockris, "Electrochemical Constants," National Bureau of Standards Circular 524, p. 243 (1953).

(4) S. G. Young (Unpublished Data, Metals Research Laboratories, Haynes Stellite Co., Division of Union Carbide Corp.).

(5) A. L. Geary (Unpublished Data, Metals Research Laboratories, Haynes Stellite Co., Division of Union Carbide Corp.).

(6) M. Stern, "Electrochemical Behavior, Including Hydrogen Overvoltage in Acid Environments," *Journal*, Electrochemical Soc., Vol. 102, p. 609 (1955).

(7) H. Kaesche and N. Hackerman, "Corrosion Inhibition by Organic Amines," *Journal*, Electrochemical Soc., Vol. 105, p. 191 (1958).

(8) M. Stern and R. M. Roth, "The Anodic Behavior of Iron," *Journal*, Electrochemical Soc., Vol. 104, p. 390 (1957).

(9) K. F. Bonhoeffer and W. Jena, "On the Electrochemical Behavior of Iron," *Zeitung für Elektrochemie*, Vol. 55, p. 151 (1951).

(10) Werner Stumm (Unpublished Data, Division of Engineering and Applied Physics, Harvard University, Cambridge, Mass.).

(11) M. Stern, "Ferrous-Ferric Electrode Kinetics on Stainless Steel," *Journal*, Electrochemical Soc., Vol. 104, p. 559 (1957).

(12) M. Stern, "The Relation between Pitting Corrosion and the Ferrous-Ferric Oxidation-Reduction Kinetics on Passive Surfaces," *Journal*, Electrochemical Soc., Vol. 104, p. 600 (1957).

(13) M. Stern, "Surface Area Relationships in Polarization and Corrosion," *Corrosion*, Vol. 14, p. 329 (1958).

(14) T. Kobayashi and M. Nagayama, "An Electrochemical Testing Method of Corrosion Inhibitors in Neutral Salt Solution," *Corrosion Engineers* (Japan), Vol. 7, p. 225 (1958).

(15) H. J. Engell, "A Method for Measurement of Corrosion Rate in Solutions Containing Air," *Archiv für das Eisenhüttenwesen*, Vol. 29, p. 553 (1958).

DISCUSSION

MR. H. J. ENGELL[1] (*presented in written form*).—The authors are to be complimented on the excellent method they developed for the rapid measurement of corrosion rates. The advantage of this method, compared with conventional long-time exposure test, is quite obvious. Starting from the same point of view, we developed[2] a very similar technique for the measurement of corrosion rates of metals in solutions containing air such as natural water, sea-water, etc. In this case the cathodic reaction is controlled by diffusion of oxygen. Then β_c approaches infinity as already pointed out by the authors. By our method not only the linear polarization resistance at the open-circuit potential, but also the curvature of the polarization curve is measured. From both values β_a can be calculated. Therefore the accuracy of our method will be better than that of the authors' technique. On the other hand, our method is limited to a special (but very common) type of corrosion reactions, in contrary to the authors' method.

MESSRS. M. STERN AND E. D. WEISERT (*authors' closure*).—Little comment can be made about Mr. Engell's excellent work. As indicated, his method is limited to cases where concentration polarization is controlling and he is to be commended for his use of measured kinetic parameters to obtain corrosion rates under these conditions. The method which we describe is, of course, not limited in this respect and, as illustrated, the accuracy of the estimate which can be made by our method is determined by the degree of knowledge of the kinetic parameters involved. If no information is available, except that the system is controlled by the concentration polarization, then our method can measure corrosion rates accurately to within about 40 per cent, with no other knowledge of the system. Indeed, if we have the necessary kinetic parameters available, our method is capable of accuracy with all systems comparable to Mr. Engell's results with systems controlled by concentration polarization.

---

[1] Max-Planck Institut für Eisenforschung, Düsseldorf, West Germany.

[2] H. J. Engell, "Method of Measurement of Dissolution Velocities of Corroding Metals in Solutions Containing Air," *Archiv für das Eisenhüttenwesen*, Vol. 29, p. 553 (1958).

THE TENSILE PROPERTIES OF SOME ENGINEERING MATERIALS AT HIGH RATES OF STRAIN*

BY ARTHUR L. AUSTIN[1] AND ROBERT F. STEIDEL, JR.[1]

SYNOPSIS

An experimental method to determine the dynamic tensile properties of materials at high rates of strain has been developed. An explosive-impact tension tester which has provisions for direct measurements of load, impact velocity, and instantaneous changes of diameter as a function of time is used. A charge of gunpowder is used to propel a projectile which is threaded to one end of a standard tension specimen, the other end being fixed.

The fracture strengths, per cent reduction of area, and per cent elongation for SAE 1018 cold-rolled steel, 6061-T6 aluminum alloy, and C120-AV, A110-AT, and A55 titanium alloys have been obtained for strain rates up to 22,000 per sec at room temperature. It is concluded that in general the dynamic strength and ductility of these materials increase with increasing strain rate.

For most engineering materials, dynamic strength increases with increasing strain rate and decreases with increasing temperature. Under the adiabatic conditions of high-speed plastic deformation experienced in impact loading, the effects of strain rate and temperature act simultaneously. The resultant effect, however, is that the dynamic strength of materials generally increases with strain rate.

In most of the previous research on the subject, the fracture has been achieved by such methods of load application as a rotating flywheel, a pendulum, or dropping a weight. Clark and Dätwyler (**1**)[2] used an Izod pendulum machine to produce strain rates to 150 per sec. Manjoine and Nadai (**2, 3**), using a rotary impact machine, produced dynamic stress-strain curves at strain rates up to 1000 per sec and at temperatures up to 1000 C. Extensive tests by Duwez, Wood, and Clark, (**4, 9**) were carried out with a rotary impact machine on long wires and standard tension specimens of varying lengths at velocities up to 200 ft per sec. In all this work involving direct mechanical impact, excessive vibrations at the higher speeds limited the range of the apparatus. Of particular interest is the work of Shepler (**10**) who used an explosive-impact tension tested to eliminate this problem. He reported strain rates as high as 25,000 per sec, but stated that due to lack of instrumentation capable of instantaneous strain measurements, his calculations were only an approximation.

It should be noted that results of all research have consistently verified that the dynamic strength of most materials increases with increasing strain rate. The purpose of the present work is to determine experimentally the effects of strain rate on various engineering materials at room temperature and to provide a means of extending the range of accurate measurements of the instantaneous strain rate.

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

[1] Lawrence Radiation Laboratory, University of California, Livermore, Calif.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

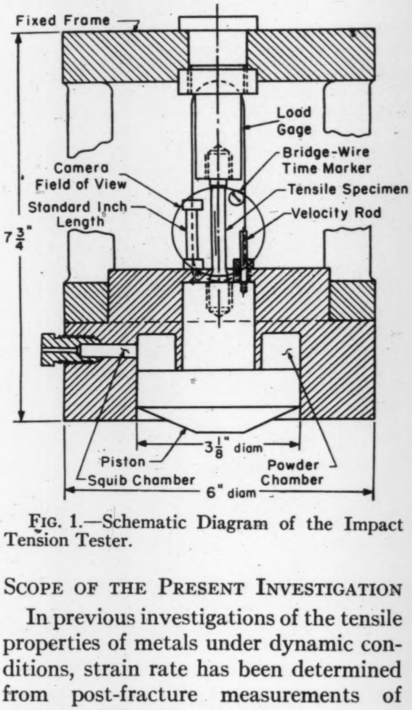


FIG. 1.—Schematic Diagram of the Impact Tension Tester.

SCOPE OF THE PRESENT INVESTIGATION

In previous investigations of the tensile properties of metals under dynamic conditions, strain rate has been determined from post-fracture measurements of strain and the average velocity or total time from yield to fracture. Since the rate of change of length is not uniform over the length of a ductile specimen during necking, it is necessary to measure instantaneous incremental length or diameter changes at the neck for an accurate determination of strain rate. In the past, lack of adequate instrumentation has always prevented such measurements.

An explosive-impact tension tester is designed somewhat after that used by Shepler (10), but with basic modifications to allow accurate correlation and simultaneous measurement of rate of change of diameter and load for tests on a standard 1-in. tension specimen.[3] A high-speed framing camera is used to measure diameter changes with the necessary accuracy. During necking of the specimen, instantaneous strain rates as high as 25,000 per sec are accurately measured over time intervals as short as 5.9 microseconds. This is some 100 times shorter than intervals attained in previous research on the subject.

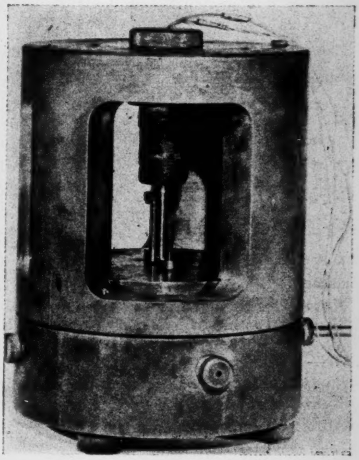


FIG. 2.—The Impact Tension Tester.

EXPERIMENTAL APPARATUS AND PROCEDURE

The Impact Tension Tester and Related Equipment:

The impact tester is composed of four main parts: a fixed frame, a steel-weigh-bar mounting for two strain gages to provide load measurements, a cylinder block, and a piston. The tester is shown schematically in Fig. 1, and Fig. 2 is an over-all photograph. One end of the specimen (Fig. 3) is threaded to the load gage and the other end to the piston, which is propelled at high speeds by a charge of gunpowder ignited in the powder chamber. The powder charge weight is adjusted to obtain the desired piston speed or impact velocity.

---

[3] Tentative Methods of Tension Testing of Metallic Materials (E 8 – 57 T), 1958 Book of ASTM Standards, Part 3, p. 108.

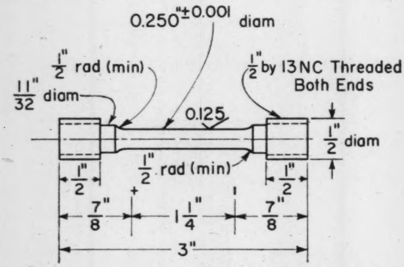


FIG. 3.—Standard ½-in. Round Tension Test Specimen.

NOTE: Design taken from Tentative Methods of Tension Testing of Metallic Materials (E 8 – 57 T), 1958 Book of ASTM Standards, Part 3, p. 108.

Surface to be polished to give indicated finish with no tool marks.

All dimensions given in inches.

For protection of the operator while loading and arming, the tester is mounted inside a firing stand constructed of a piece of 12-in. pipe 24 in. long and having the necessary openings for photographing the event. The piston is fired downward into a large box of sand as shown in Fig. 4. The combined weight of the sand and firing stand prevents movement of the tester and affords the required fixed base. All of the firing equipment is housed inside a concrete bunker (Fig. 5), fitted with a mirror arrangement to allow the camera to view the event from an adjoining room.

Instrumentation:

Load measurement is provided by two A-5 resistance-wire strain gages mounted diametrically opposite on the load cell (Fig. 6). To eliminate bending and measure only axial load, the gages are connected in series in one leg of the Wheatstone bridge circuit in a model BA-12 Ellis Associates bridge amplifier. A 240-ohm compensating gage completes the other external leg of the bridge. The output of the bridge amplifier is fed to a 535 Textronic oscilloscope fitted with a 53/54D preamplifier. The oscilloscope trace is recorded with a type 2614 Dumont Polaroid-Land camera loaded with type 44 film. A static calibration of the load gage provides conversion of the trace to a load-time curve.

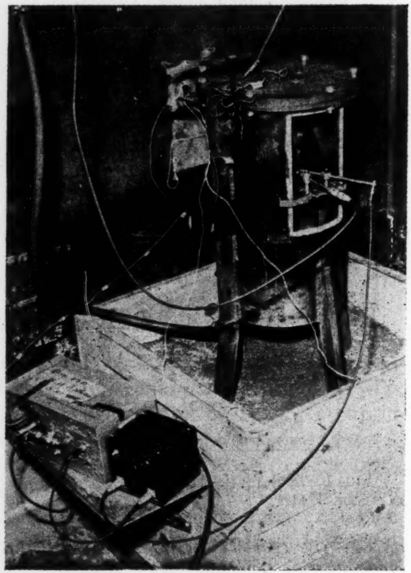


FIG. 4.—Impact Tension Tester and Related Equipment.

A model 602 high-speed framing camera (developed at Los Alamos Scientific Laboratory) fitted with a 30-in. lens and operating at 500 rps (5.9 microseconds interframe time) is used to record impact velocity and rate of re-

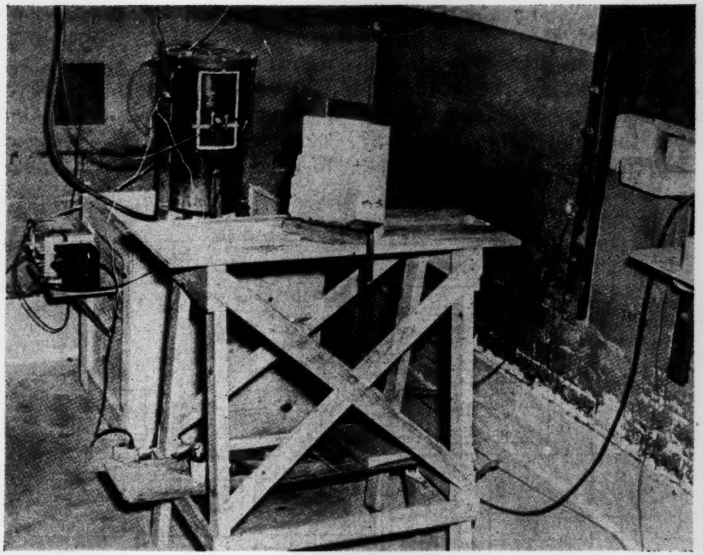


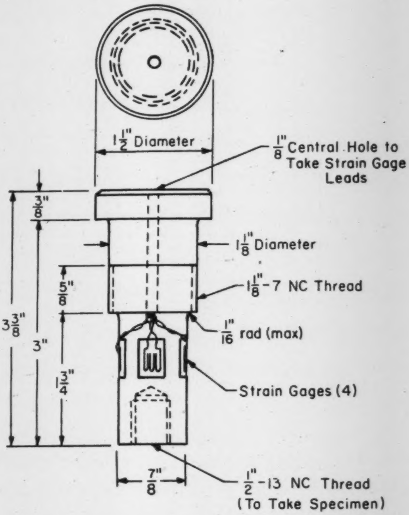
FIG. 5.—Test Cell Showing Camera Portal and Mirror.

duction of diameter of the specimen undergoing deformation to fracture. Since ignition of the powder charge varies from 15 to 70 milliseconds, unsynchronized operation of the camera is required. Silhouette lighting of the specimen is provided by a single GE FT-220 electronic-flash unit modified to give a maximum effective flash duration of 350 microseconds.

Hercules Bullseye pistol powder is used as the propellent. It is a violent powder with a very fast pressure rise, which provides excellent control of piston velocity with only a small change in charge weight. One Hercules blasting squib is used for ignition and is fired with the condenser detonation unit provided in the camera-control rack.

Synchronization of Events:

The schematic block diagram (Fig. 7) and the function-time diagram (Fig. 8) show the synchronization and operation



$1\frac{1}{2}$" Diameter
$\frac{1}{8}$" Central Hole to Take Strain Gage Leads
$\frac{3}{8}$"
$1\frac{1}{8}$" Diameter
$\frac{5}{8}$"
$1\frac{1}{8}$"-7 NC Thread
$3\frac{3}{8}$"
3"
$\frac{1}{16}$" rad (max)
$1\frac{3}{4}$"
Strain Gages (4)
$\frac{7}{8}$"
$\frac{1}{2}$"-13 NC Thread (To Take Specimen)

FIG. 6.—Load Cell Showing Placement of SR-4 Strain Gages. Material: SAE 1020 Mild Steel.

NOTE: All dimensions in inches, tolerances omitted.

of the apparatus. After the tester is loaded and armed, the strain-gage circuitry balanced, and the trigger circuits checked, the bunker is darkened and the camera is loaded with 35-mm Tri-X film. When the rotor is brought up to speed (500 rps), the squib is fired and the powder charge drives the piston downward. The piston movement closes the first trigger circuit which starts the oscilloscope sweep. Further piston movement closes the second circuit, which triggers the flash lamp and causes exposure of the film strip. Since the camera shutter is manually held open for unsynchronized operation, the flash duration must be short enough to prevent rewriting.

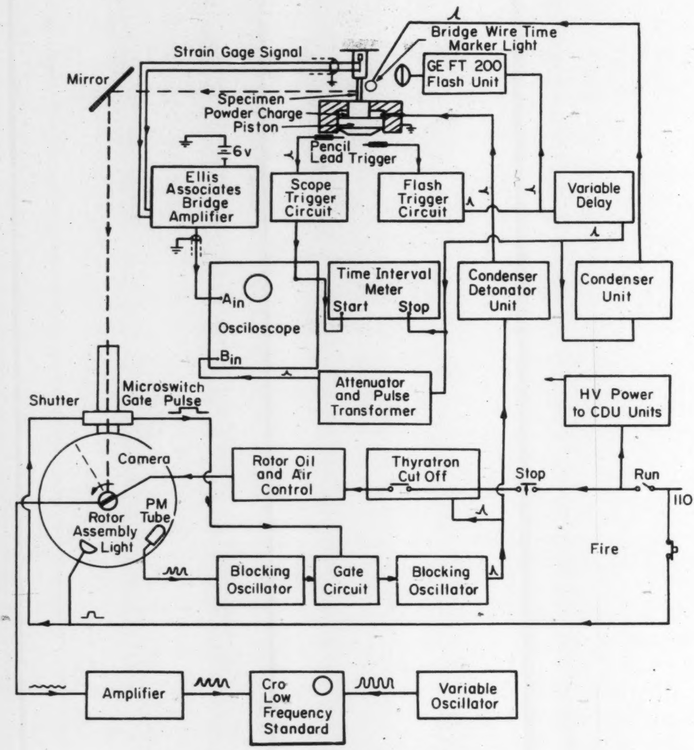

Bridge Wire Time Marker Light
Strain Gage Signal
Mirror
GE FT 200 Flash Unit
Specimen
Powder Charge
Piston
Pencil Lead Trigger
6v
Ellis Associates Bridge Amplifier
Scope Trigger Circuit
Flash Trigger Circuit
Variable Delay
Time Interval Meter
Start
Stop
Condenser Detonator Unit
Condenser Unit
A in
Osciloscope
B in
Attenuator and Pulse Transformer
HV Power to CDU Units
Shutter
Microswitch Gate Pulse
Camera
Rotor Oil and Air Control
Thyratron Cut Off
Stop
Run
110v
Rotor Assembly
PM Tube
Light
Fire
Blocking Oscillator
Gate Circuit
Blocking Oscillator
Amplifier
Cro Low Frequency Standard
Variable Oscillator

FIG. 7.—Schematic Block Diagram.

The oscilloscope trigger is preset to record as much of the load-time trace as possible, and the flash trigger is set to operate at the expected elongation of the specimen where necking will start. The trigger circuits are shown in Fig. 9.

Correlation of the load-time trace with the diameter-time record is accomplished by using the flash-trigger pulse to fire a bridge-wire mounted next to the specimen. This marks a particular frame in time. The pulse is also superimposed as a pip on the oscilloscope trace and provides a common time reference for both records. A Beckman model 7360 time-interval meter is used as a check should the bridge-wire fail to fire.

strip with a scanning microscope, and load is measured directly from the Polaroid photograph of the oscilloscope trace. Sample film strips and oscilloscope traces are shown in Figs. 10, 11, and 12.

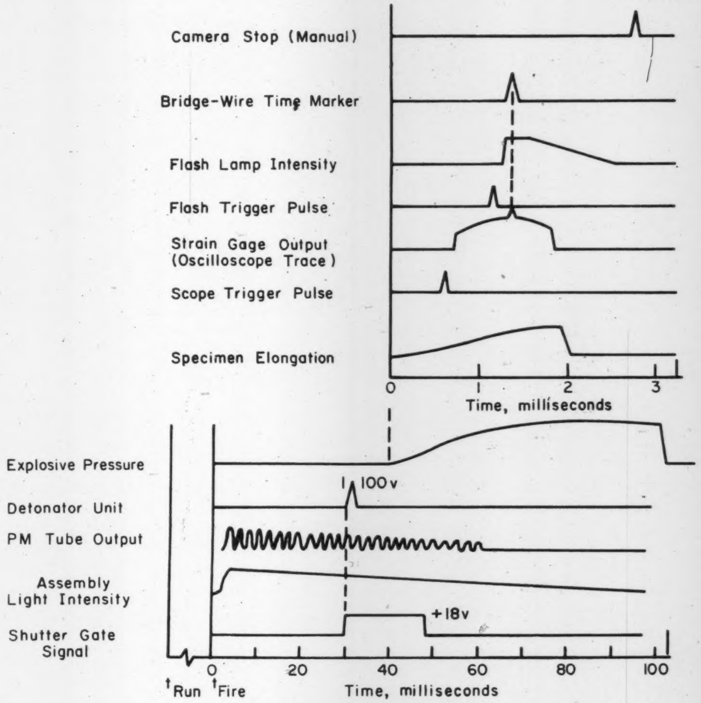

Camera Stop (Manual)
Bridge-Wire Time Marker
Flash Lamp Intensity
Flash Trigger Pulse
Strain Gage Output (Oscilloscope Trace)
Scope Trigger Pulse
Specimen Elongation
0
1
2
3
Time, milliseconds
Explosive Pressure
Detonator Unit
100v
PM Tube Output
Assembly Light Intensity
+18v
Shutter Gate Signal
0
20
40
60
80
100
Run
Fire
Time, milliseconds

FIG. 8.—Function-Time Diagram.

Measurements:

The bar of known length on the left in the camera field of view provides calibration for film-strip measurements. The velocity rod on the right moves with the piston and provides measurement of piston velocity or impact velocity. In the upper right, the bridge-wire fiducial is shown. The changes of diameter of the specimen are measured from the film

Piston displacement, diameter, and load are plotted on a common time axis as shown by the example in Fig. 13. Impact velocity and strain rate are determined by graphical measurement of the slope of the curves at a point halfway between the start of necking and fracture. Average true stress is obtained by dividing load by the corresponding area. The following equations are used:

$$\text{Strain rate, } e = \frac{2 \ln(D_1/D_2)}{t_2 - t_1} \text{ in. per in. per sec} \ldots (1)$$

$$\text{Impact velocity, } v = \frac{h_2 - h_1}{t_2 - t_1} \text{ ft per sec} \ldots (2)$$

where:

D_1, D_2 = diameter, in., $D_1 > D_2$,
h_1, h_2 = piston displacement, ft, $h_1 < h_2$, and
t_1, t_2 = time, sec, $t_1 < t_2$.

DISCUSSION OF RESULTS

The results of the tests on SAE 1018 cold-rolled steel and 6061-T6 aluminum alloy in Figs. 14 and 15 show that the fracture strengths increase continuously with increasing strain rate up to 24,000 per sec. Also, the per cent reduction of area and per cent elongation indicate that the ductility of these materials approaches a constant value with increasing strain rate.

Corrected fracture-stress curves have been drawn in as dotted lines. The correction factor is obtained from a calculation by Bridgman (11) who has shown that the stress at the neck of a tension specimen consists of a uniform axial stress across the section with a superimposed hydrostatic tension which varies from zero at the surface to a maximum at the center. As shown, the amount of correction remains nearly constant with strain rate and is approximately 10 per cent for steel and 14 per cent for aluminum alloy.

Consideration must be given to the fact that for all curves the plotted points do not represent direct measurements of strain rate but instead are calculated values taken from graphical measurements of the slopes of the diameter-time curves. These slopes are taken at a point halfway between the start of necking and fracture where the strain rate is a maximum. In order to be consistent, the fracture stress should be plotted *versus* the strain rate exactly at fracture. At this point, however, the strain rate will necessarily be zero since diameter change must be zero for fracture to occur. The per cent reduction of area and per cent elongation represent direct measurements from the broken specimen.

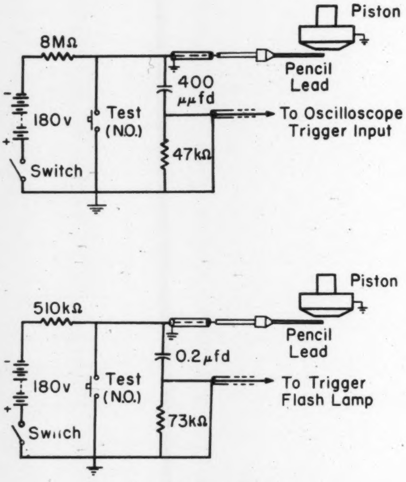


FIG. 9.—Trigger Circuits.

The results of tests on C120-AV and A110-AT titanium alloys are shown in Figs. 16 and 17. For A110-AT alloy the fracture stress decreases to a minimum at approximately 11,000 per sec and continuously increases thereafter. At this point the "ultimate" strength rises to a maximum and is nearly equal to the fracture stress. It is interesting to note that per cent reduction of area and per cent elongation are a minimum at this point. The data are questionable for fracture stress at strain rates of 10,000

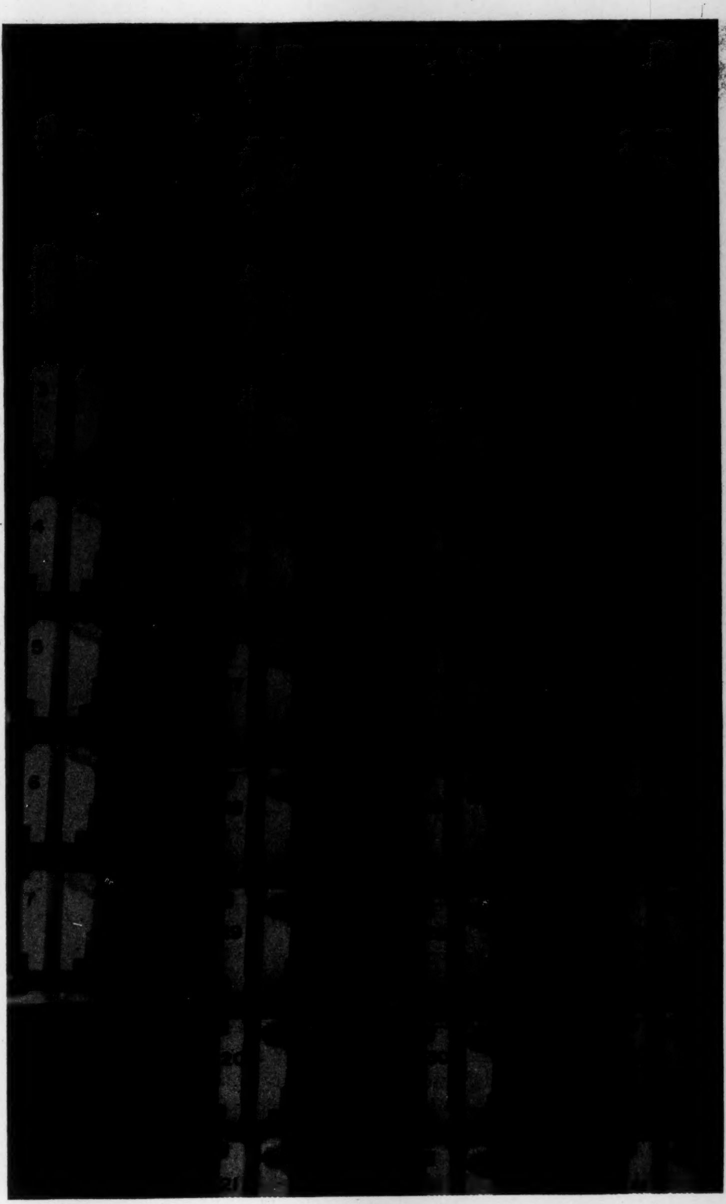
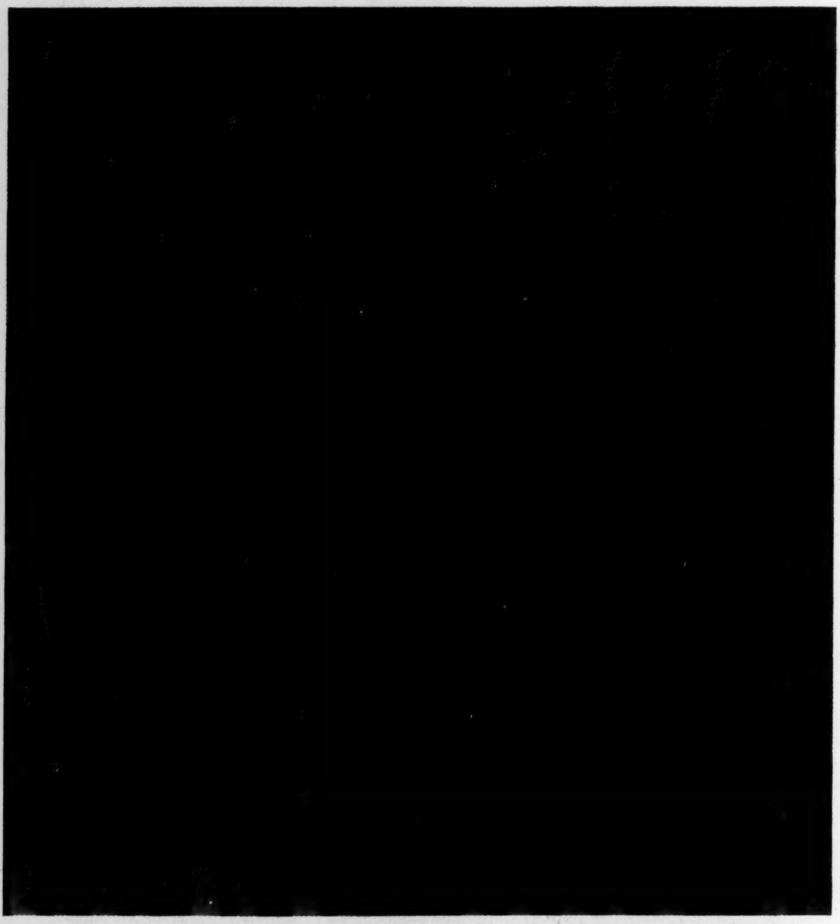


FIG. 10.—Sample Film Strips.
6061-T6 aluminum alloy; 5.9 microseconds per frame.



(*a*) No. 5.—C120-AV titanium alloy. (*b*) No. 6.—A110-AT titanium alloy.
FIG. 11.—Sample Film Strips.

per sec and 13,900 per sec. The oscilloscope traces for these two runs are very faint, and consequently the fracture loads are not clearly defined. This is the only apparent explanation for these deviations.

For C120-AV alloy the fracture stress and "ultimate" strength follow the same trend as that for the A110-AT alloy, but ductility does not minimize. The per cent reduction of area increases continuously with strain rate, and per cent elongation remains constant.

Measurement of true stress at the neck of a tension specimen under dynamic conditions is subject to many errors. The measurement of load at the fixed end of the specimen is only the

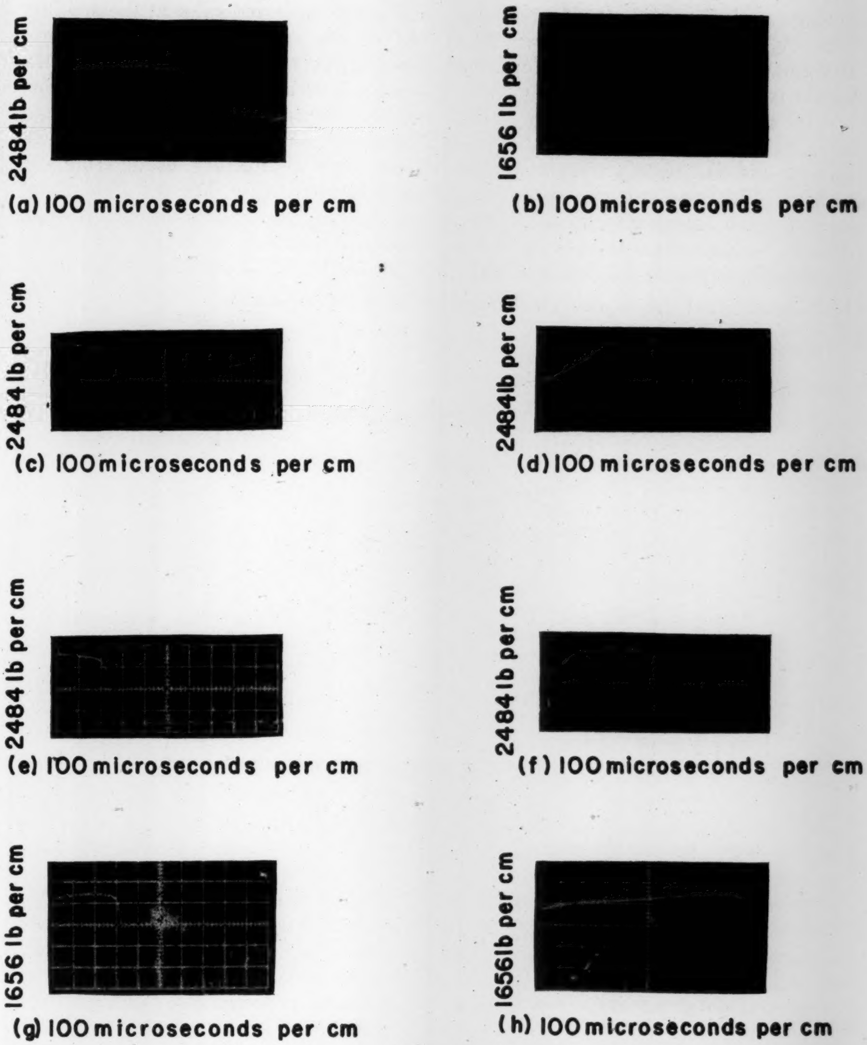


FIG. 12.—Oscilloscope Traces.

(*a*) No. 52.—SAE 1018 cold-rolled steel.
(*b*) No. 57.—6061-T6 aluminum alloy.
(*c*) No. 5.—C120-AV titanium alloy.
(*d*) No. 8.—C120-AV titanium alloy.
(*e*) No. 6.—A110-AT titanium alloy.
(*f*) No. 8.—A110-AT titanium alloy.
(*g*) No. 3.—A55 titanium alloy.
(*h*) No. 2.—A55 titanium alloy.

average load since the actual stress distribution at the neck is not known. The Bridgman correction aids in accounting for the triaxial state of stress at the neck, but for the titanium alloys it could not be applied. The titanium-alloy cross-sections did not remain circular during necking. This also introduces an error in strain-rate measurement since it is difficult to determine which diameter is recorded in silhouette on the film strip. It is found that the major and minor diameters differ by as much as 0.047 in. This results in an error of about 16 per cent for strain rates calculated from Eq 1. An approximation of the neck cross-section by an ellipse with major and minor axes obtained from post-fracture measurements of the specimen is used for fracture-stress calculations.

The measurements of load at the fixed end of the specimen are corrected for the effects of the dynamic forces by assuming that the acceleration of the plastic mass increases linearly from zero at the fixed end to the measured value at the moving end of the specimen. This correction, however, is approximately 5 per cent. Since it is within the error of reading the oscilloscope traces, it is usually neglected. Examination of the traces in Fig. 12 will illustrate the difficulty in determining the load at fracture. For the aluminum-alloy trace, the fracture point is well defined in contrast to the trace for SAE 1018 steel, where a 10 to 15 per cent deviation is possible.

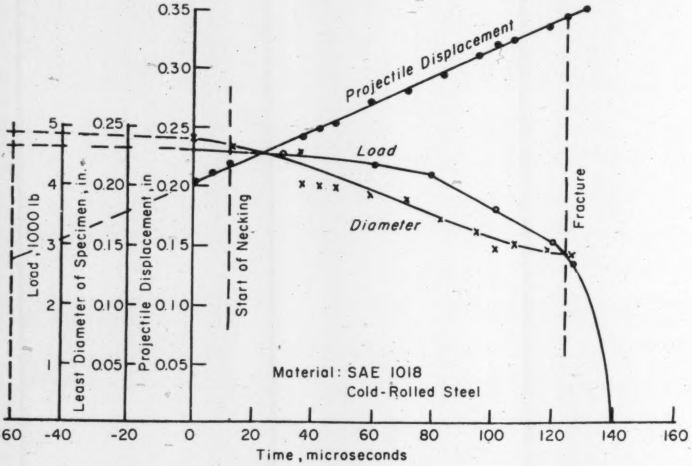


FIG. 13.—Graph Showing Common Time Axis on Which Piston Displacement, Diameter and Load Are Plotted.

The curves for commercially pure A55 titanium (Fig. 18) show that reduction of area reaches a minimum at a strain rate of 9000 per sec with elongation continuously decreasing. Fracture stress remains nearly constant to a strain rate of approximately 6000 per sec and increases thereafter.

Examples of fractured specimens are shown in Fig. 19. All specimens fractured in the manner shown, with large reductions of areas. The steel, aluminum, and A55 titanium specimens all maintained

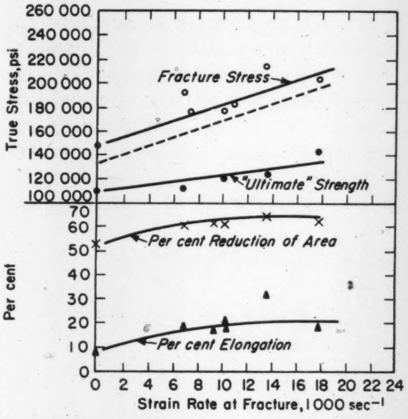


FIG. 14.—Explosive-Impact Tension Tests at Room Temperature.

Material: SAE 1018 cold-rolled steel.

Standard ASTM 0.250 in. diameter × 1.000 in. gage length tensile specimen.[3]

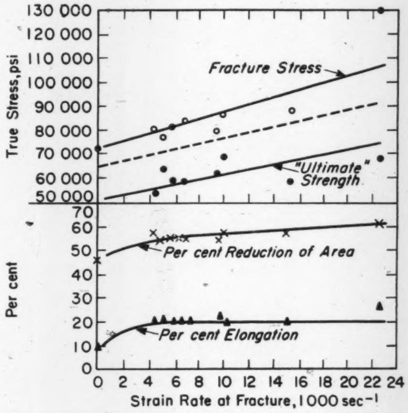


FIG. 15.—Explosive-Impact Tension Tests at Room Temperature.

Material: 6061-T6 aluminum alloy.

Standard ASTM 0.250 in. diameter × 1.000 in. gage length tensile specimen.[3]

circular cross-sections during necking to fracture. The C120-AV and A110-AT titanium-alloy specimens did not remain circular, but necked down into approximate elliptical cross-sections with eccentricities as high as 0.6. This is probably caused by the preferred orientation induced by the initial working of the raw material.

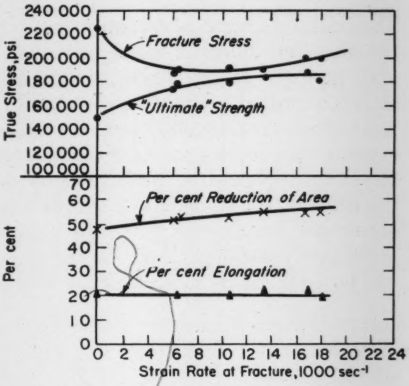


FIG. 16.—Explosive-Impact Tension Tests at Room Temperature.

Material: C120-AV titanium alloy.

Standard ASTM 0.250 in. diameter × 1.000 in. gage length tensile specimen.[3]

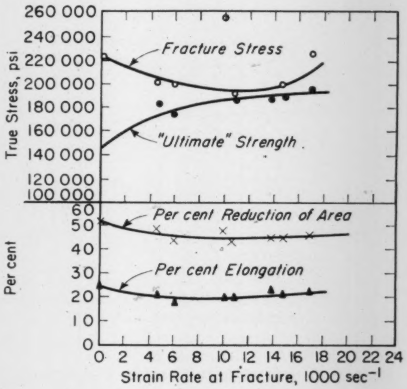


FIG. 17.—Explosive-Impact Tension Tests at Room Temperature.

Material: A110-AT titanium alloy.

Standard ASTM 0.250 in. diameter × 1.000 in. gage length tensile specimen.[3]

It should be noted that the curves in Figs. 14–18 are arbitrarily chosen as the best representation of the plotted points.

The initial decrease of fracture stress of the titanium alloys is merely assumed, and perhaps these curves should be shown as dotted over the range of strain rates from zero to 4000 per sec. It is evident that more tests in this range of strain rates should be performed. With the present tester, the lowest strain rate obtainable for ductile materials is approximately 4000 per sec. Lower rates can be achieved by increasing the piston weight and decreasing the powder charge. For this work, powder charges are varied from 2 to 20 g.

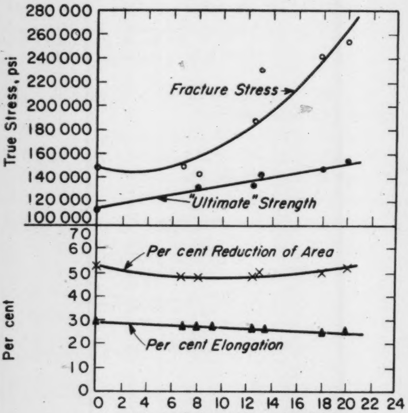


FIG. 18.—Explosive-Impact Tension Tests at Room Temperature.

Material: A55 commercially pure titanium. Standard ASTM 0.250 in. diameter × 1.000 in. gage length tensile specimen.[3]

From the results of tests reported here, it is evident that the dynamic behavior of materials cannot be predicted from static tension tests alone. Although dynamic testing may not simulate actual conditions, it does provide an intelligent comparison of materials for use in a particular dynamic application. The explosive-impact tester provides a means of evaluating the dynamic properties of materials over a wide range of strain rates and, in addition is adaptable for consideration of various parameters.

It is planned that future work in dynamic testing will be directed toward the study of the properties of materials as a function of strain rate with parameters of temperature, type or rate of change of loading, and material heat treatment. Impact tension tests will be performed at elevated temperatures with various load-time functions obtained by using different types of propellents.

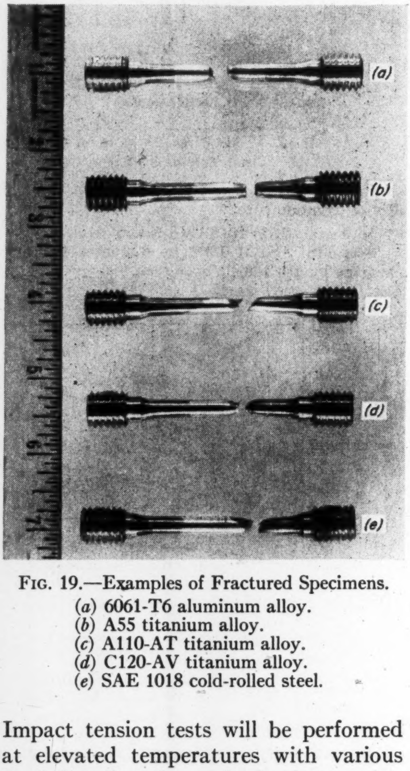


FIG. 19.—Examples of Fractured Specimens.
(*a*) 6061-T6 aluminum alloy.
(*b*) A55 titanium alloy.
(*c*) A110-AT titanium alloy.
(*d*) C120-AV titanium alloy.
(*e*) SAE 1018 cold-rolled steel.

It has been found in all of the work on the dynamic behavior of materials that the dynamic properties are markedly dependent upon material heat treatment. For future work, the prime consideration will be the parameter of heat treatment.

TABLE I.—TABULATION OF RESULTS.

Material	Part Number	Strain Rate at Fracture, per sec	Fracture Stress, 1000 psi	Tensile Strength, 1000 psi	Reduction of Area, per cent	Elongation, per cent	Impact Velocity at Fracture, ft per sec
SAE 1018 cold-rolled steel....	Static	0	148	110	45.6	8.0	0
	48	6 920	193	112	60.5	15.3	71.1
	51	7 360	177	120	61.5	19.3	73.5
	52	10 000	178	120	62.0	20.6	100.0
	..	10 800	182	...	....	18.0	92.0
	49	13 500	215	125	64.0	31.3	146.0
	50	17 700	204	144	62.0	17.1	143.0
6061-T6 aluminum alloy.....	55	4 480	80	54	58.0	19.9	58.0
	45	5 130	77	64	54.2	20.7	38.5
	56	5 850	82	59	56.3	20.0	57.5
	43	6 890	84	58	55.0	20.4	63.0
	47	7 130	...	...	55.2	20.5	62.5
	57	9 550	79	61	55.0	21.8	77.0
	44	10 000	87	69	56.3	20.3	87.5
	58	15 400	88	58	57.6	20.2	90.0
	59	22 600	131	68	61.3	25.5	162.2
	Static	0	72	50	46.8	25.5	0
Titanium alloy C120-AV, 6 per cent aluminum, 4 per cent vanadium, 0.1 per cent carbon..................	Static	0	225	150	46.9	20.0	0
	9	6 420	189	176	51.9	....	40.6
	1	6 500	191	179	52.5	19.2	.....
	5	10 700	194	180	52.0	18.6	.....
	6	13 460	190	187	54.8	21.5	.....
	3	17 000	200	189	54.0	19.7	.....
	8	18 100	201	181	54.7	16.5	.....
Titanium alloy A110-AT, 5 per cent aluminum, 2.5 per cent tin..................	Static	0	225	144	51.0	25.0	0
	10	4 650	205	183	48.0	20.8	53.8
	9	6 050	200	174	44.4	17.8	38.7
	8	10 000	255	182	47.5	19.3	115.0
	2	10 900	191	185	43.4	19.5	96.8
	7	13 900	229	186	44.8	23.0	128.0
	6	14 740	198	188	45.0	27.0	68.3
	5	17 100	226	195	46.2	22.0	143.0
Titanium alloy A55, commercially pure titanium....	Static	0	150	112	52.6	29.8	0
	2	6 800	150	148	48.2	26.1	63.7
	1	8 000	144	133	48.2	27.5	34.3
	4	9 100	...	...	49.8	27.1	136.0
	3	12 500	189	134	48.2	26.4	104.0
	5	13 000	231	142	50.4	26.7	163.0
	6	18 000	241	148	50.4	24.7	198.0
	7	20 000	254	156	52.1	25.3	206.0

CONCLUSIONS

1. The dynamic fracture strength of SAE 1018 cold-rolled steel and 6061-T6 aluminum alloy increases continuously with increasing strain rates up to 22,000 per sec. The per cent reduction of area and per cent elongation of these materials increase continuously with strain rate, but tend to approach a constant value at the higher rates.

2. The dynamic fracture strength of C120-AV and A110-AT titanium alloys decreases to a minimum at a strain rate of 11,000 per sec and continually increases thereafter. A corresponding minimum in per cent reduction of area and

per cent elongation occurs for A110-AT titanium alloy, and for C120-AV the per cent reduction of area increases continuously with per cent elongation, remaining constant for strain rates up to 18,000 per sec.

3. The dynamic fracture strength of commercially pure A55 titanium increases strain rate. Per cent reduction of area minimizes at a strain rate of 8000 per sec and per cent elongation continually decreases.

REFERENCES

(1) D. S. Clark and G. Dätwyler, "Stress Relations Under Tension Impact Loading," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, pp. 98–111 (1938).

(2) M. J. Manjoine and A. Nadai, "High-Speed Tension Tests at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 822 (1940).

(3) M. J. Manjoine and A. Nadai, "High-Speed Tension Tests at Elevated Temperatures," *Transactions*, Am. Soc. Mechanical Engrs., *Journal Applied Mechanics*, June 1941, p. A77.

(4) D. S. Clark, "The Influence of Impact Velocity on the Tensile Characteristics of Some Aircraft Metals and Alloys," *Technical Note 868*, Nat. Advisory Committee for Aeronautics, Oct. 1942.

(5) P. E. Duwez, D. S. Clark and D. S. Wood, "The Influence of Specimen Length on Strain Propagation in Tension," *NDRC Report 1-105*, Office of Scientific Research and Development, No. 957.

(6) P. E. Duwez, D. S. Clark, and D. S. Wood, "The Influence of Impact Velocity on the Tensile Properties of Plain Carbon Steel and of a Cast Steel Armor Plate," *NDRC Report A-154*, Office of Scientific Research and Development, No. 1274.

(7) P. E. Duwez, D. S. Clark, and D. S. Wood, "The Effect of Stopped Impact and Reflection on the Propagation of Plastic Strain in Tension," *NDRC Report A-108*, Office of Scientific Research and Development, No. 988.

(8) P. E. Duwez, D. S. Clark, and D. S. Wood, "Dynamic Tests of the Tensile Properties of SAE 1020 Steels, Armco Iron, and 17ST Aluminum Alloy," *NDRC Report A-182*, Office of Scientific Research and Development, No. 1490.

(9) P. E. Duwez, D. S. Clark, and D. S. Wood, "Discussion of Energy Measurements in Tension Impact Measurements at the California Institute of Technology," *NDRC Report A-217*, Office of Scientific Research and Development, No. 1829.

(10) P. R. Shepler, "Impact Research," Doctor of Science thesis submitted to Mechanical Engineering Department, Massachusetts Institute of Technology, Sept. 27, 1943.

(11) P. W. Bridgman, "The Shape of the Neck and the Fracture of Tension Specimens," *Experimental Report No. WAL 111/7-4*, Watertown Arsenal Laboratory, Jan., 1944.

DISCUSSION

MR. FRANK R. LARSON[1] (*by letter*).—In 1936 and 1937, Mann (1, 2)[2] reported here at the ASTM some data that were obtained with rotating flywheel equipment. One of the significant observations from his tests was that there existed, for even materials that we normally believe to behave in a ductile manner, a critical velocity at which elongation drops off. We know from von Karman's (3, 4) work and some of the very fine work that was done at the California Institute of Technology (5) that this behavior is a result of the inability of a material to propagate a plastic wave above a certain critical velocity. This velocity is dependent upon the shape of the stress-strain curve. In this case the engineering stress-strain curve is applicable, not the true stress–natural strain curve.

I would like to ask the authors if they observed this critical velocity since their velocities were a little higher than Mann's velocities, and I do not see any evidence in these results of such a critical velocity. Analysis of the broken specimen shape would be very revealing and reporting the location of the fracture in relation to whether it is at the struck or held end of the specimen.

The data reported on the fracture stress needs to be clarified because Bridgman had shown from his work at Harvard (6) that the fracture stress or the stress in any transverse location of the bar, in the plane of maximum stress, is dependent upon the shape of the neck. In other words, from what I gather from the paper, the authors have reported something that may be more precisely termed flow stress at fracture. It is necessary to be very careful about terminology, and one of the questions that arises, assuming you want to make a proper fracture stress calculation, is that you have to know the shape of every bar and the actual location of the fracture point, because quite frequently these bars do not break at the center of the specimen where the peak hydrostatic stress exists.

Along with this, I would also like to ask the authors if they measured the geometry of these broken specimens (shape of the neck), so that one could arrive at some study of what the fracture stress was in terms of whether it is the shear stress, normal stress, or the peak hydrostatic tension at the location where the fracture initiated. A great deal more detailed study is needed, even in a simple test like a tension test, to actually pin down what is the status of the stress at the fracture point, at least in some gross scale. I do not believe when there are inclusion and things like that, that we are going to be able to get too good a result, but we should, I think, attempt to define the conditions a little more precisely.

MR. R. F. STEIDEL, JR. (*author's closure*).—The phenomenon observed as critical velocity occurs in an impact tension test, for instance, where one end of the specimen is caused instantaneously to move at a constant velocity. Both plastic and elastic stress waves are produced by this impact, the elastic front gradually moving ahead of the plastic wave due to the higher velocity of the former. The velocity of propagation of the plastic strain decreases in proportion to the square root of the tangent modulus of the experimental stress - strain curve. The critical velocity is represented by the inability of the material to propagate the strain along the bar in accordance with this experimental stress - strain relation.

---

[1] Physical Metallurgist, Watertown Arsenal Laboratories, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this discussion.

In the reported tests, the loading of the tensile bar does not conform to that usually associated with impact tests. The end of the specimen under explosive loading does not move instantaneously at a constant velocity, but is gradually accelerated. This gradual acceleration would mean that more of the tensile bar is being strained than in the previous case. The end velocity at which the material would be unable to propagate longitudinal strain would thus be somewhat higher. As a result, a critical velocity was not encountered in the explosive impact tests.

One of the main advantages of using photographic techniques to observe dimensional changes with time is that it provides a permanent visual record of the strain histories. For each tensile bar tested, a film record of the necking process was obtained. Examples are shown in Figs. 10 and 11 of the paper. Such records were used to obtain instantaneous measurements of neck radius, *a*, and neck radius of curvature, *R*, as a function of load. The flow stress, *F*, was then calculated from the formula

$$F\left(1 + 2\frac{R}{a}\right)\log\left(1 + \frac{1}{2}\frac{a}{R}\right)$$

$$= \frac{\text{Load}}{\pi a^2} = \text{Average tension stress}$$

derived by Bridgman (reference (11) of the paper). As pointed out in the paper, this was done for the steel and aluminum specimens only with the flow stress or "corrected fracture stress" curves shown as dotted lines (Figs. 14 and 15 of the paper). Measuring the radius of curvature as a function of load for each bar tested proved to be a slow and tedious procedure; hence, a curve of a/R *versus* $2 \log (a_0/a)$ for tests on a variety of materials by Bridgman (reference (11) of the paper) was used with sufficient accuracy to provide values for a/R rather than make entirely direct measurements of *R*.

REFERENCES

(1) H. C. Mann, "High Velocity Tension Impact Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 85 (1936).

(2) H. C. Mann, "Fundamental Study of the Design of Impact Test Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 102 (1937).

(3) Theodore von Karman, "On the Propagation of Plastic Deformation in Solids," National Defense Research Committee Report No. A-29, Office of Scientific Research and Development, No. 365, Feb. 2, 1942.

(4) Theodore von Karman and P. E. Duwez, "On the Propagation of Plastic Strains in Solids," presented at Sixth International Congress for Applied Mechanics, Sept. 1946, Paris (France).

(5) D. S. Clark and D. S. Wood, "The Tensile Impact Properties of Some Metals and Alloys," *Transactions*, Am. Soc. for Metals, Vol. 42, p. 45 (1950).

(6) P. W. Bridgman, "The Stress Distribution at the Neck of a Tension Specimen," *Transactions*, Am. Soc. for Metals, Vol. 32, p. 553 (1944).

COMPRESSION TESTING AT CONSTANT TRUE STRAIN RATES*, †

BY J. E. HOCKETT[1]

SYNOPSIS

A cam plastometer was designed and built to test metal specimens in compression over a range of constant true strain rates and over a range of temperatures. Commercially pure aluminum specimens were tested at room temperature at three strain rates. Depleted uranium specimens were tested at several strain rates and temperatures.

From measurements of load and time throughout each test, true stress *versus* true strain curves were calculated. These curves fit an equation of the form

$$\sigma = A\ (1 - e^{B\epsilon}) + C\epsilon$$

where σ is the true stress, ϵ is the true strain, A, B, and C are parameters, and e is the base of natural logarithms. The parameters A and C were found to be dependent upon temperature and strain rate, increasing with decreasing temperature and with large increases in strain rate. The parameter B was found to be essentially independent of temperature, but a large increase of strain rate produced an increase in B.

Single tests and pools of several tests, performed under identical conditions, were fitted to the above equation by a least squares method using electronic computers. Variance and standard deviation of fit of the curve, variances and standard deviations of the three parameters, variances and standard deviations of the mean stresses and of prediction of stresses were also calculated for each strain.

The use of the cam plastometer, along with the statistical methods described, provides an accurate means of investigating the effects of temperature and strain rate on the resistance of specimens to compression. The effects of alloying elements, impurities, grain size, and thermal treatments on the resistance to compression may also be determined by this method.

In order to perform calculations of the forces and pressures involved in a number of metal-forming operations it is necessary to know the yield, or flow stress over a considerable range of strains, strain rates, and temperatures. For accurate calculations these must be true stresses, true strains, and true strain rates (1).[2]

Such fabricating methods as rolling,

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

† This work is part of that submitted to the University of California, Berkeley, Calif., as a thesis, in partial fulfillment of the requirements for the degree of Doctor of Engineering. The work was performed at the Los Alamos Scientific Laboratory under the Graduate Thesis Program.

[1] Staff Member, Metallurgical Research Group CMF-13, Los Alamos Scientific Laboratory, Los Alamos, N. Mex.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

forging, and extrusion involve principally compressive stresses. Despite the usual assumption in many calculations of forming pressures, the true stress-strain curve in compression is not generally the same as the true stress-strain curve in tension (2); hence, the use of data obtained in tension testing tends to introduce errors into these calculations.

Because the true strains attainable in a properly conducted compression test are greater than those at which necking usually occurs in a tension test and because it was desired to use the results in forming calculations involving compressive stresses, it was decided to conduct a series of compression tests at various temperatures and strain rates. However, it is extremely difficult to control the true strain rate in conventional testing machines, and the entire range of strain rates available in the usual equipment is considerably lower than the rates encountered in most forming operations. It was therefore deemed advisable to build a machine to compress metallic specimens over a range of strain rates extending into the higher rates encountered in such operations as rolling, for example.

A machine, called a cam plastometer, was designed and built at Los Alamos Scientific Laboratory for the above purpose. The basic principle of the cam plastometer was first proposed by Orowan (3) and employed in a small machine built at the British Iron and Steel Research Assn. laboratories. This principle involves a logarithmic cam which applies the compressive load to the test specimen at a constant true strain rate which is controlled by the speed of rotation of the cam.

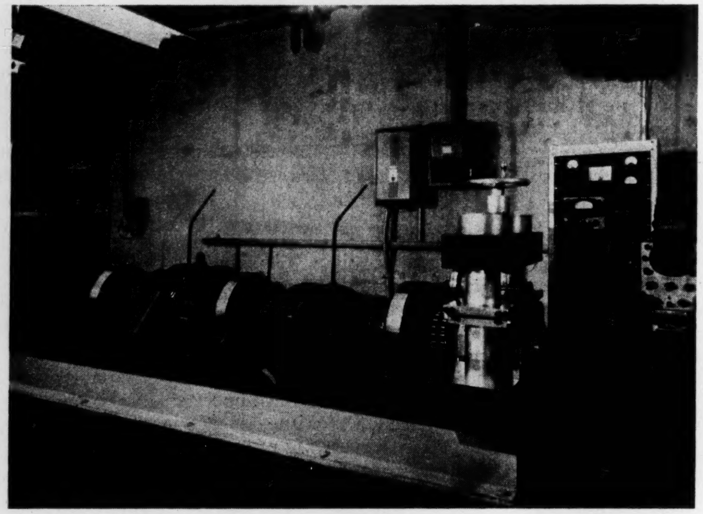


FIG. 1.—The Cam Plastometer.

A number of compression tests were performed on commercially pure aluminum specimens at room temperature as well as on normal uranium specimens (depleted of part of the uranium-235 present) at several temperatures.

Measurements of load and time

throughout each test were used to compute true stress *versus* true strain experimental points. These points were found to fit an empirical equation quite well. A least squares method was used to fit the experimental points to the equation and to smooth the resulting true stress *versus* true strain curve. The laborious and time-consuming calculations that this curve fitting required were performed in minutes on electronic computers. The parameters in the empirical equation were investigated as to temperature and rate dependence, and such dependence was determined.

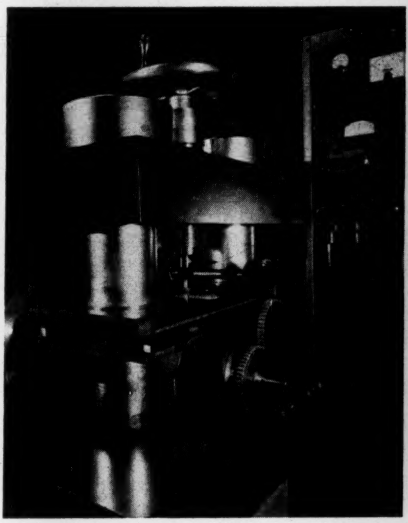


FIG. 2.—Aluminum Specimen in Plastometer Press Prior to Compression Test.

THE CAM PLASTOMETER—DESCRIPTION AND OPERATION

An over-all view of the cam plastometer at Los Alamos is shown in Fig. 1. An aluminum specimen is shown in Fig. 2 in position for a test. The press end of the plastometer, as it appears during elevated temperature tests, is shown in Fig. 3. Visible are the heating controls, potentiometer for measuring specimen temperatures, inert gas tube, and recorder.

The logarithmic cam was designed to compress 0.800-in. specimens to 50 per cent of their original height in one half of a revolution. The true strain rate is constant so long as the cam speed remains constant.

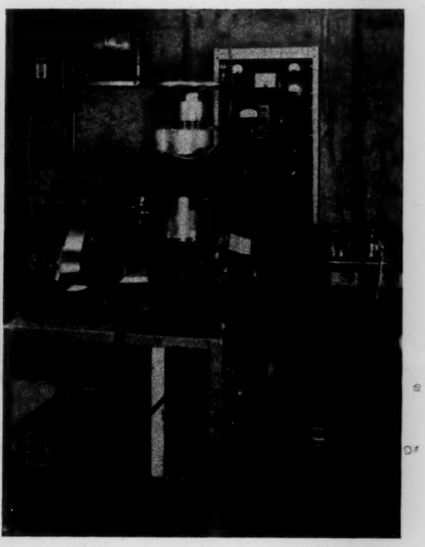


FIG. 3.—Press End of Plastometer Prior to Elevated Temperature Test.

The cam is driven by a 50-hp d-c slightly compounded motor through a drive train consisting of three truck transmissions and three 20-in. flywheels. Power is supplied to the motor at 125 v by a 40-kw motor-generator set operating on 440-v alternating current. The combination of three transmissions in series and the d-c motor provides a wide range of cam speeds and, consequently, a wide range of strain rates.

Operation of the plastometer consists of bringing the cam to the desired speed, inserting a cam-following slide between the cam and a riser block, compressing the specimen, and removing the cam-following slide. The cam-following slide is inserted mechanically, by a series of gears and linkage arms, during the one half revolution when the cam is cylindrical. During the next half-revolution, the cam lifts the slide, the riser block above it, and the tungsten carbide platen resting in the riser block. This upward movement compresses the specimen against an upper carbide platen which, in turn, is forced against a load cell. The slide drops at the end of compression, and it is removed to a position away from the cam during the next half-revolution when the cam is again cylindrical, thereby preventing any subsequent lifting of the riser block and lower platen.

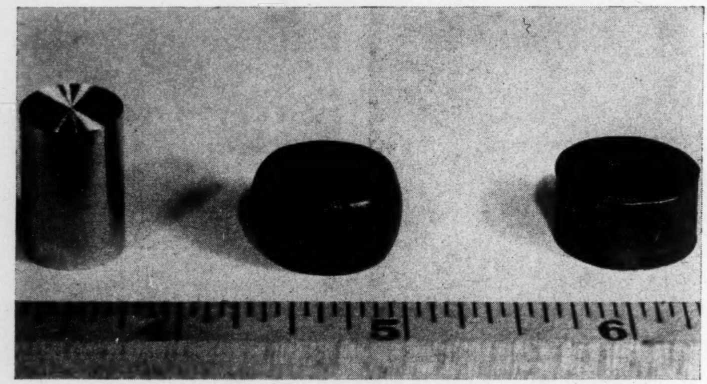


FIG. 4.—Depleted Uranium Compression Specimens. Left: prior to test; Center: after test with improper lubricant; Right: after test with proper lubricant.

Because of inertia in the mechanical method of inserting and removing the cam-following slide, the plastometer was restricted to relatively low speeds, so that the range of true strain rates was about 10^{-3} per sec to 1 per sec. However modifications under way are expected to increase the maximum rate to about 2.5 × 10 per sec.

PROBLEMS IN TESTING

The principal difficulty in compression testing is the elimination of the effects of friction between the specimen ends and the platens. Unless friction is virtually eliminated, deformation will not be uniaxial but will be inhomogeneous, as evidenced by "barreling" of the specimen. These end effects were alleviated by a method proposed by Orowan (3) and used by Loizou and Sims (4) and by Alder and Phillips (5). Concentric grooves were machined in the specimen ends, suitable lubricants were used, and the tungsten carbide platens were ground and hand-lapped with diamond paste to an rms No. 2 finish. The depth, spacing, and included angle of the concentric grooves were found to be critical. A groove spacing of 74 per in., a groove depth of 0.004 in., and an included angle of about 64 deg were found suitable for uranium specimens. Two different powdered glasses were used as lubricants at 600 and at 500 C. From 400 C to room temperature, minus 200 mesh powdered molybdenum disulfide suspended in machine oil was satisfactory. Figure 4 shows an electropolished specimen ready for testing, a specimen after testing with an inadequate lubricant, and a specimen after testing with a suitable lubricant. The glasses used at 600 and 500 C, respectively, were: (1) 27.3 per cent by weight SiO_2, 71.0 per cent by weight PbO, 1.5 per cent by weight K_2O; and (2) 20.0 per cent by weight B_2O_3, 80.0 per cent by weight PbO.

Other problems encountered in this work were control of temperature uniformity and surface oxidation at elevated temperatures. A heater unit was developed which permitted control of temperature to ±5 C at all parts of the specimen when testing at 600 C. Oxidation was reduced to a bare minimum by maintaining a positive pressure of helium gas in the heater unit. The heater is shown in Fig. 5.

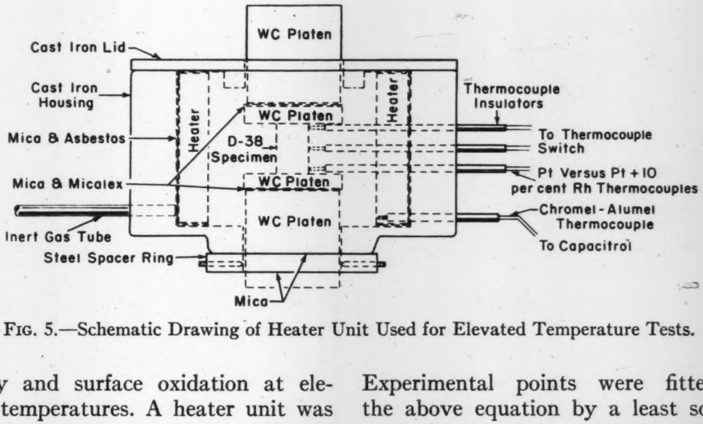

Cast Iron Lid
WC Platen
Cast Iron Housing
Mica & Asbestos
Heater
WC Platen
D-38 Specimen
Mica & Micalex
WC Platen
WC Platen
Heater
Thermocouple Insulators
To Thermocouple Switch
Pt Versus Pt + 10 per cent Rh Thermocouples
Chromel - Alumel Thermocouple
To Capacitrol
Inert Gas Tube
Steel Spacer Ring
Mica

FIG. 5.—Schematic Drawing of Heater Unit Used for Elevated Temperature Tests.

TEST RESULTS

Specimens of commercially pure aluminum were tested at room temperature at constant true strain rates of 2×10^{-1}, 4.6×10^{-1}, and 1.5 per sec. Specimens of depleted uranium were tested at 600, 500, 400, and 300 C, at constant true strain rates of about 10^{-3}, 10^{-1}, and 1 per sec. It was found that experimental true stress-strain data gave a reasonable fit in an empirical equation of the form:

$$\sigma = A(1 - e^{B\epsilon}) + C\epsilon \qquad (1)$$

where:

σ = true stress,
ϵ = true strain,
A, B, and C = parameters, and
e = base of natural logarithms.

Experimental points were fitted to the above equation by a least squares method adapted from Deming (6), and described in the Appendix.

A typical true stress-strain curve for a compression test of depleted uranium at a constant true strain rate of 1.146×10^{-1} per sec, at 500 C is shown in Fig. 6. The experimental points, the smoothed curve, and the standard deviation are shown.

The results of tests on aluminum are shown in Table I, and those for depleted uranium in Table II. For each strain rate the values are for an average of all experimental points from the number of tests indicated. In Table I

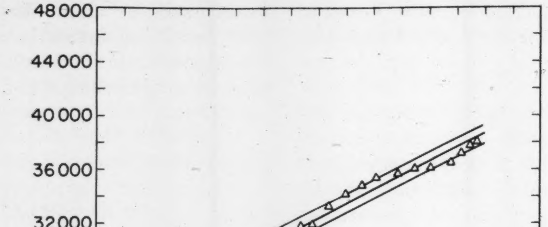


FIG. 6.—Typical True Stress-Strain Test on Uranium at 500 C. $\dot{\epsilon} = 1.146 \times 10^{-1}$ per sec.

TABLE I.—RESULTS OF COMPRESSION TESTS ON COMMERCIALLY PURE ALUMINUM AT CONSTANT TRUE STRAIN RATES.

Data fit to equation: $\sigma = A(1 - e^{B\epsilon}) + C\epsilon$

Strain Rate, $\dot{\epsilon}$, per sec	Variance of Fit, s_f^2	Standard Deviation of Fit, s_f	First Parameter, A	Standard Deviation of A, s_A	Second Parameter, B	Standard Deviation of B, s_B	Third Parameter, C	Standard Deviation of C, s_C	Number of Points, n	Number of Tests
2.30×10^{-1}....	347 103	589.2	11 303	237	−25.92	1.46	13 496	473	70	4
4.55×10^{-1}....	2 496 161	1 579.2	11 662	637	−19.12	2.54	13 220	1 221	89	5
1.46..........	2 749 815	1 685.3	10 972	515	−22.49	2.80	15 750	1 037	124	7

it can be seen that varying the strain rate over the short range investigated with aluminum produced no significant effects on the parameters of Eq 1. However, for uranium, decreasing the temperature increased the values of the parameters *A* and *C*, as did increasing the strain rate. The parameter *B*, for uranium, appears to be relatively independent of temperature, but a large increase in strain rate, for example, from 10^{-3} to 1 per sec, seems to increase the value of *B*.

SUMMARY

The compression testing method described, using the cam plastometer, provides an accurate and reproducible method of determining the effects of true strain rate and temperature on the resistance of metals to compression.

The statistical methods employed, although tedious, are of great help (1) in determining the accuracy of a group of tests, that is, the discrepancy or scatter between a number of tests conducted under identical conditions; and (2) in designing future experiments.

TABLE II.—RESULTS OF COMPRESSION TESTS ON DEPLETED URANIUM AT CONSTANT TRUE STRAIN RATES.

Data fit to equation: $\sigma = A(1 - e^{B\epsilon}) + C\epsilon$

Strain Rate, $\dot{\epsilon}$, per sec	Temperature, deg. Cent	Variance of Fit, s_f^2	Standard Deviation of Fit, s_f	First Parameter, A	Standard Deviation of A, s_A	Second Parameter, B	Standard Deviation of B, s_B	Third Parameter, C	Standard Deviation of C, s_C	Number of Points, n	Number of Tests
1.41×10^{-3}	600	900 939	949.2	8 606	336	−19.33	1.62	5 044	702	124	7
1.12×10^{-1}	600	4 820 246	2 195.5	13 781	583	−35.41	3.95	7 865	1 322	129	9
1.14	600	3 361 636	1 833.5	14 106	531	−32.50	3.27	8 234	1 191	105	7
2.07×10^{-3}	500	2 000 018	1 414.2	16 622	435	−19.28	1.13	16 811	905	168	8
1.09×10^{-1}	500	19 657 575	4 433.7	23 950	949	−34.36	3.59	20 433	2 184	205	9
1.10	500	19 484 078	4 414.1	26 662	837	−33.40	2.85	15 723	1 965	234	10
1.52×10^{-3}	400	6 152 080	2 480.3	28 504	687	−19.37	1.01	29 609	1 475	214	6
0.743	400	27 570 034	5 250.7	38 560	1108	−29.01	2.00	29 228	2 698	243	7
1.24×10^{-3}	300	6 032 344	2 456.1	40 723	545	−21.07	0.61	43 804	1 181	301	6

Further experiments have been proposed, and will, in fact, be conducted as soon as modifications to the plastometer designed to extend the range of strain rates have been completed. It is intended to investigate further the effects of strain rate and temperature on the resistance to compression of the relatively pure depleted uranium used in this work. Then, it is intended to ascertain the effects of alloying elements, impurities, thermal treatments, and grain size on the compressive strengths of uranium and other metals.

Acknowledgments:

The author wishes to express his appreciation for the counsel and guidance of Mr. Earl R. Parker, of the University of California, in this investigation. Gratitude is also due Mr. Robert B. Gibney and Mr. Morton C. Smith of the Los Alamos Scientific Laboratory for their advice and encouragement.

In addition, the author appreciates the use of the facilities of the Los Alamos Scientific Laboratory and the opportunity to conduct the experiments, perform the calculations, and publish the results under the Graduate Thesis Program at Los Alamos.

APPENDIX

LEAST SQUARES FITTING OF STRESS-STRAIN CURVES

The general problem of fitting experimental points to a curve, the equation of which may contain a number of parameters, is described by Deming (6). He derives the general solution, based upon the theory of propagation of error, and gives several illustrations of its use. Hence, the derivation will not be given here. The application of this method to the fitting of experimental points to Eq 1, smoothing of the resultant true stress *versus* true strain curve, and calculation of the different variances and standard deviations follows:

Approximate values of the three parameters, A, B, and C, are calculated and substituted in Eq 1. Values of $\Delta\sigma_i$, the differences between calculated and experimental values of stress for a number of values of strain, ϵ_i, are calculated. These values are used in the reduced normal equations, which can be represented by a four-by-three matrix. The matrix is solved in the standard way, that is, by forward and backward solutions, to obtain new values of the parameters. Then new values of the $\Delta\sigma_i$, or "residuals" are computed, the matrix solved again, and the process is repeated until the difference between the nth solution and the $(n - 1)$ solution divided by the nth solution, for each parameter, is equal to or less than 10^{-6}. This may require as few as two or three, or as many as fifteen or twenty repetitions. Then the reduced normal equations are solved three more times to obtain the inverse matrix.

The best estimate, s_f^2, of the variance of "fit" of the curve, σ_f^2, is obtained from:

$$s_f^2 = \frac{\sum_{i=1}^{n} \Delta\sigma_i^2}{K} \quad \text{............} (2)$$

where:

$K = n - p$,

n = the number of points,

p = the number of parameters, and

$\Delta\sigma_i$ = the residuals.

The variances of the parameters are obtained from:

$$s_A^2 = C_{11}\, s_f^2 \quad \text{...........} (3a)$$

$$s_B^2 = C_{22}\, s_f^2 \quad \text{...........} (3b)$$

$$s_C^2 = C_{33}\, s_f^2 \quad \text{...........} (3c)$$

where the C_{ij} are specific values along the diagonal of the inverse matrix.

The "variance of the mean" stress, for a given strain, from a population of stresses, which are assumed to be distributed normally about the mean stress is estimated by:

$$s_{\sigma m}^2 = \left[\sum_{i=1}^{3}\sum_{j=1}^{3}\left(\frac{\partial F}{\partial A_i}\right)\left(\frac{\partial F}{\partial A_j}\right) C_{ij}\right] s_f^2 \quad \text{..} (4)$$

where:

F = the function $[A(1 - e^{B\epsilon}) + C\epsilon_i]$,

A_i and A_j = values of the parameters A, B, and C $(i = 1$ to 3, $j = 1$ to $3)$,

C_{ij} = values from the inverse matrix,

and

s_f^2 = the variance of fit of Eq 2.

The variance of prediction of the true value of the stress, for a given strain—which is different than the mean of Eq 4—is estimated by:

$$s_{\sigma p}^2 = \left[\sum_{i=1}^{3}\sum_{j=1}^{3}\left(\frac{\partial F}{\partial A_i}\right)\left(\frac{\partial F}{\partial A_j}\right) C_{ij} + 1\right] s_f^2 \quad . (5)$$

where the terms have the same meaning as in Eq 4.

The above calculations were coded for computation on IBM 704 Electronic Data Processing Machines. Running time for the problem was from 2 to 4 min, depending upon the number of points and the number of repetitions of the matrix solution.

REFERENCES

(1) W. S. Wagner, "Plastic Working of Metals," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 78, No. 2, p. 401 (1956).

(2) E. Voce, "True Stress-Strain Curves, and Their Application to Cold-Working Processes," *Metal Treatment*, Vol. 15, No. 54, pp. 53–66, 72 (1948).

(3) E. Orowan, "The Cam Plastometer," British Iron and Steel Research Assn., Report MW/F/22/50 of the Forging Committee in the Mechanical Working Division, March, 1950.

(4) N. Loizou and R. B. Sims, "The Yield Stress

of Pure Lead in Compression," *Journal of Mechanics and Physics of Solids*, Vol. 1, pp. 234–243 (1953).

(5) J. F. Alder and V. A. Phillips, "The Effect of Strain Rate and Temperature on the Resistance of Aluminum, Copper, and Steel to Compression," *Journal*, Inst. of Metals, Vol. 83, pp. 80–86 (1954).

(6) W. E. Deming, "Statistical Adjustment of Data," John Wiley and Sons, Inc., New York, N. Y., (1946).

DISCUSSION

MR. C. W. MACGREGOR[1] (*presented in written form*).—In presenting compression-test data obtained under conditions of constant true-strain rates, the author has contributed new and useful information of a type greatly needed. About 14 yrs ago, the writer and his colleagues presented similar information through true-stress-strain tension tests conducted at constant true-strain rates (1).[2] The rates varied from 5×10^{-5} to 5×10^{-3} sec^{-1} and were conducted at various temperatures, both low and high, on SAE 1045 and 1020 steels as well as on brass. The true-strain rates were chosen so that no test would be made rapidly enough to produce a change in temperature by internal heating and so that no test would last more than 6 hr. They were thus conducted under isothermal conditions. The highest strain rate was a little higher than the lowest strain rates reported by the author. The writer would like to ask if in the author's tests any appreciable change in temperature occurred due to internal heating during loading. At elevated temperatures the interrelationship of strain rate and temperature is a sensitive one.

It is possible that the author has somewhat overemphasized the errors present in using tension test data of the true-stress-strain type in metal forming calculations where compression stresses predominate. The difference between flow stresses for the two tests is relatively small for ductile metals, especially under the elevated-temperature conditions commonly employed. Recent studies (2) showed that quite accurate predictions of contact rolling pressure distributions as well as rolling loads and torques could be made utilizing true-stress-strain tension data. In most metal forming problems, such unknown items as friction and the relatively crude assumptions made to facilitate a solution are of much greater effect on the final result than slight differences between tension and compression test data. Nevertheless, it is useful to have compression data of the type submitted.

It is also mentioned that it is extremely difficult to control the true strain rates in conventional testing machines. While it is more difficult to conduct such tests than the more conventional ones, the writer found no particular problem in carrying this out at normal speeds. With two operators, and with a preselected schedule of diameters at definite times, such tests are conducted by manually varying the platen speeds to produce and maintain these strain rates. At more rapid speeds, special devices would be required.

For more rapid speeds, however, it is possible to avoid making actual tests to determine the information required, and thus to avoid such concomitant problems as inertia, traveling wave effects, and internal heating under uncontrolled conditions. Use can be made of the velocity-modified temperature (3) concept to determine stress reactions at higher speeds and different temperatures. This was suggested and utilized by the writer and his colleagues to predict results under wide changes of strain rate from relatively slow-speed tests. Data such as are contained in Table II of the paper for depleted uranium are of the type needed. The velocity-modified temperature T_m is

---

[1] Consulting Engineer, Philadelphia, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this discussion.

$$T_m = T\left[1 - k \ln \frac{\dot{\epsilon}}{\dot{\epsilon}_0}\right]$$

where:

T = the testing temperature in degrees absolute,
$\dot{\epsilon}$ = the corresponding true-strain rate,
$\dot{\epsilon}_0$ = a base strain rate, and
k = a material constant.

A value of k may be determined from the data (3) and the stress reaction plotted as a function of T_m for any given strain. The data in Table II should fall along a single smooth curve for each value of the true strain selected. These curves may then be used to determine the stress reaction for wide changes of temperature and strain rate.

The writer would like to congratulate the author on his paper and urge him to continue this useful study.

MR. J. E. HOCKETT (*author's closure*).—The author appreciates Mr. MacGregor's comments on the paper and will attempt to answer them.

The author is quite familiar with the work described by the discusser (1) and appreciates the value of isothermal testing. However, because the test results, such as those in Table II of the paper, were intended for use in forming calculations—specifically rolling calculations—an attempt was made to duplicate the strain rates and temperatures encountered in rolling uranium. To answer the question about specimen temperature, there undoubtedly was a temperature rise in the specimen during deformation, as there is during rolling. Recent tests on Armco iron at room temperature confirmed that the temperature does rise appreciably during high-speed compression tests. Rough calculations, involving some assumptions, indicate the specimen temperature could rise as much as 80 to 100 C in a high-speed test at room temperature.

The work on Armco iron indicates that the temperature rise during a test masks the strain rate effect to some extent. It was suggested, or implied, in the discussion that the concept of a velocity-modified temperature, as derived in Mr. MacGregor's references (1,3), might compensate for this masking and enable one to predict stress reactions at high speeds from the results of low-speed tests.

The velocity-modified temperature, as originally presented, was derived from the Eyring (4) Kauzman (5) Dushman (6) theory and appeared in the discusser's reference (3) as:

$$S = f\left[T\left(1 - K \ln \frac{\dot{\epsilon}}{\dot{\epsilon}_0}\frac{T_0}{T}\right)\right]$$

where the symbols are as indicated in the discussion, S is the stress, T_0 is a constant, and the expression in brackets is T_m. Mr. MacGregor considered that the range of temperatures investigated in creep testing was small. In fact, he assumed the ratio of maximum temperature to minimum temperature to be unity, that is, $T_{\max}/T_{\min} \cong 1$ for creep. Therefore, T_0 was chosen so that $T_0/T \cong 1$. In the paper, and in more recent work, this ratio $(T_{\max}/T_{\min})$ varied up to a factor of 3.5, and T_0/T could not be taken as unity.

A laborious attempt was made to fit the detailed data of Table II to the original Dushman (6) equation. They did not fit. This should preclude, then, any chance of fitting the data to the velocity-modified temperature concept that was derived from Dushman's work.

However, because a number of simplifying assumptions were made in the derivation of the velocity-modified temperature, it was considered possible that the concept might apply to these data. A number of attempts were made to utilize the velocity-modified temperature. Stress was found to be a nonlinear function of temperature over a range of strains from 0.1 to 0.7 for several strain rates. Hence, values of K and $\dot{\epsilon}_0$ were not immediately obvious. Ranges of values of K and $\dot{\epsilon}_0$ were tried in attempts to handle the data using the velocity-modified temperature. Plots of T_m *versus* S did not yield smooth curves in any instance. It should be pointed out that a number of attempts were made to fit the data of Table II to empirical equations. The best fit was to the equation used in this work. This equation reflects the effects of changes in temperature and strain rate by changes in the parameters A and C, and this is at the temperatures and strain rates of interest, for example, those actually obtained during the rolling of uranium.

With regard to flow stresses in compression *versus* flow stresses in tension, the difference becomes significant when roll pressure calculations are performed by Orowan's "exact" theory (7), which is what was done with the results in Table II (8). In these calculations, essentially no simplifying assumptions are made. The coefficient of friction between rolls and material rolled must be known accurately, but this can be determined at the temperatures of interest.

The author appreciates Mr. MacGregor's encouragement. Further work of this nature is planned and is, in fact, under way at present.

REFERENCES

(1) C. W. MacGregor and J. C. Fisher, "Tension Tests at Constant True Strain Rates," *Journal of Applied Mechanics*, Vol. 12, No. 4, Dec. 1945, pp. A217–A227.

(2) C. W. MacGregor and R. B. Palme, "The Distribution of Contact Pressures in the Rolling of Metals," *Paper 58-A-260*, presented at the 1958 Annual Meeting of the Am. Soc. Mechanical Engrs. (*to be published*).

(3) C. W. MacGregor and J. C. Fisher, "A Velocity-Modified Temperature for the Plastic Flow of Metals," *Journal of Applied Mechanics*, March 1946, pp. A11-16.

(4) H. Eyring, "Viscosity, Plasticity, and Diffusion as Examples of Absolute Reaction Rates," *Journal of Chemical Physics*, Vol. 4, pp. 283–291 (1936).

(5) W. Kauzman, "Flow of Solid Metals from the Standpoint of the Chemical-Rate Theory," *Metals Technology*, Technical Publication No. 1301, Am. Inst. Mining and Metallurgical Engrs., June, 1941.

(6) S. Dushman, L. W. Dunbar, and H. Huthsteiner, "Creep of Metals," *Journal of Applied Physics*, Vol. 15, pp. 108–124 (1944).

(7) E. Orowan, "The Calculation of Roll Pressures in Hot and Cold Flat Rolling," *Proceedings*, Inst. Mechanical Engrs., Vol. 150, pp. 140–167 (1943).

(8) J. E. Hockett, "The Rolling Pressures of Uranium Sheet and Plate," *Report LA-2233*, Los Alamos Scientific Laboratory, Office of Technical Services, U. S. Department of Commerce, Washington, D. C.

COMPRESSION TESTS OF WOOD AT ELEVATED TEMPERATURES

By S. H. Ingberg[1]

This paper presents information on the effect of elevated temperatures on the compressive strength of two species of wood used for structural purposes. With initial moisture content of 7 to 9 per cent, typical for wood well-seasoned indoors, there was a loss in strength at 106 C of about 50 per cent of the strength at room temperature, with strains 50 to 100 per cent greater. At temperatures in the range 151 to 162 C there was regain in strength to about 70 per cent of that at room temperature, due apparently to shrinkage and hardening from further loss of water. A final decline in strength was indicated at temperature near 200 C. With loads maintained for longer periods, the reduction in strength was greater. With the heat insulation given specimens in the testing equipment, there was no sustained self-heating of wood at temperatures up to 208 C. In tests with end-point temperatures of 279 and 294 C such self-heating caused nearly full loss of strength from destructive distillation of the wood.

Materials and Specimens

The specimens were of Southern longleaf pine or Douglas fir with properties of the wood given in Table I. The density is based on weight and dimensions of specimens taken immediately before testing. The density of the Southern pine specimens was above the average for the species, while that for the Douglas fir was near the average. The grain of the wood was largely parallel with the length of the specimens, all but a few of which were free from knots and checks. The dimensions of the specimens are given in Fig. 1. They were cut from kiln-dried stock and stored under room conditions for 3 months to more than a year before testing. The moisture content at the time of testing was apparently within the range 7 to 9 per cent, as will be later indicated under Loss in Weight.

Equipment and Method

The same equipment was used as in previous tests with structural steel and cast iron (**1**).[2] It consisted essentially of a hydraulic system for applying the load and a heavily insulated alundum furnace tube extending 5 in. beyond the ends of the specimen, with two sets of end-compensating heating coils over or beyond the ends.

Deformation during test was measured by observing with microscopes the movement of fine heat-resistant alloy wires attached at each end of the 5$\frac{3}{4}$-in. gage length, the lower ends of which were attached to weights immersed in oil to dampen vibrations. The microscopes were mounted in micrometer slides on an invar bar secured to a system of slides to enable positioning and refocusing without moving the microscopes in their tubes.

Two methods of testing were used, one

---

[1] **Structural and Fire Protection Engineer Bethesda, Md.; Former Chief, Fire Resistance Section, National Bureau of Standards.**

[2] **The boldface numbers in parentheses refer to the list of references appended to this paper.**

with constant temperature and the other with constant load. With the one, the temperature was held constant and the load increased to failure; with the other, a load was applied and held constant and the temperature increased to failure.

TABLE I.—PROPERTIES OF WOOD IN SPECIMENS.

Test	Species	Density, g per cu cm	Rings, per inch	Summer-wood, per cent	Notes
No. 1	Southern pine	0.66	23	40	
No. 2	Southern pine	0.66	28	...	
No. 3	Southern pine	0.63	22	40	
No. 4	Southern pine	0.76	25	40	
No. 5	Southern pine	0.82	20	55	
No. 6	Southern pine	0.73	32	35	
No. 7	Southern pine	0.74	24	50	
No. 8	Southern pine	0.77	21	55	
No. 9	Southern pine	0.66	23	35	One fine check along most of length
No. 10	Douglas fir	0.49	18	30	
No. 11	Douglas fir	0.51	20	25	
No. 12	Douglas fir	0.54	12	35	
No. 13	Douglas fir	0.52	21	30	One ¼-in. knot
No. 14	Douglas fir	0.55	8	35	
No. 15	Douglas fir	0.48	4	30	
No. 16	Douglas fir	0.48	15	30	
No. 17	Douglas fir	0.50	9	30	Small knot at one end
No. 18	Douglas fir	0.51	16	35	
No. 19	Southern pine	0.72	27	50	
No. 20	Southern pine	0.82	25	50	
No. 21	Southern pine	0.70	22	45	
No. 22	Douglas fir	0.54	11	35	
No. 23	Douglas fir	0.51	14	30	

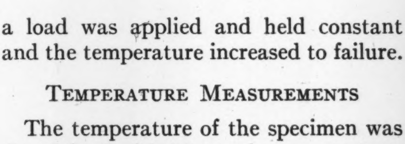


FIG. 1.—Dimensions of Specimen.

TEMPERATURE MEASUREMENTS

The temperature of the specimen was determined with 18-gage iron-constantan thermocouples connected to a potentiometer. Three were located in the hole at the center line of a specimen—one at the center and one near each end of the gage length—insulated from each other and the end bearing blocks with asbestos sleeving and mineral wool. Three were placed on the surface of the specimen in corresponding positions, their junctions being covered with $\frac{1}{16}$-in. asbestos sleeving. Near the end of the test the temperature uniformity along the gage length was within 5 C. In the cross-section the difference at failure between the average at the center and on the surface ranged from 2 to 13 C, except that in three constant-load tests the range was from 37 to 65 C. In the constant-load tests it was difficult to predict the temperature at which failure would occur and apply a rate of temperature rise to give the desired degree of uniformity at failure.

The average effective temperature for the cross-section was taken as 0.45 times the average for the center plus 0.55 times the average for the surface. Straight-line temperature variation from the center to the surface would give a space average equal to one third of the center temperature plus two thirds of the surface temperature. For parabolic temperature variation, with the vertex of the parabola at

TABLE II.—RESULTS OF TESTS AT CONSTANT TEMPERATURES.

Test,	Species	Density, g per cu cm	Average Temperature of Specimen, deg Cent	Compressive Strength, psi	Part of Derived Room-Temperature Strength
No. 1	Southern pine	0.66	Room	8 350	...
No. 2	Southern pine	0.66	Room	7 800	...
No. 3	Southern pine	0.63	Room	7 210	...
No. 4	Southern pine	0.76	Room	10 220	...
No. 5	Southern pine	0.82	Room	11 650	...
No. 6	Southern pine	0.73	106	4 340	0.45
No. 6	Southern pine	...	Room	6 750[a]	0.71
No. 7	Southern pine	0.74	106	4 910	0.51
No. 8	Southern pine	0.77	151	5 550	0.53
No. 9	Southern pine	0.66	162	5 630	0.70
No. 10	Douglas fir	0.49	Room	6 410	...
No. 11	Douglas fir	0.51	Room	6 770	...
No. 12	Douglas fir	0.54	Room	7 710	...
No. 13	Douglas fir	0.52	Room	7 050	...
No. 14	Douglas fir	0.55	Room	7 310	...
No. 15	Douglas fir	0.48	Room	5 640	...
No. 16	Douglas fir	0.48	106	3 230	0.55
No. 16	Douglas fir	...	Room	7 040[a]	1.18
No. 17	Douglas fir	0.50	154	5 340	0.83
No. 17	Douglas fir	...	Room	5 340[a]	0.83
No. 18	Douglas fir	0.51	205	4 200	0.62
No. 18	Douglas fir	...	Room	5 630[a]	0.83

[a] Retested after cooling to room temperature.

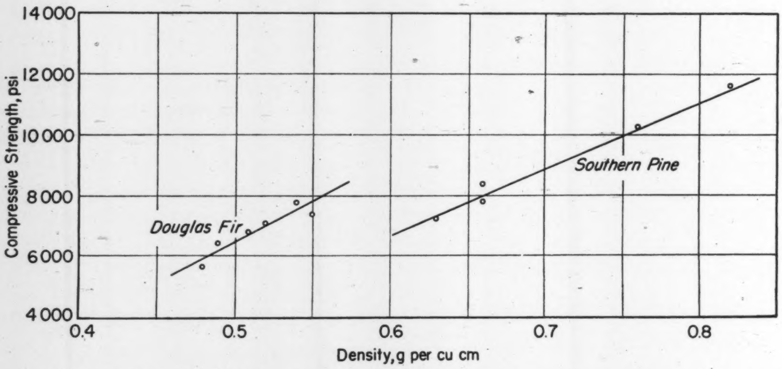


FIG. 2.—Strength-Density Relations for Tests at Room Temperature.

the center of the specimen, the space average would be equal to the average of center and surface temperatures. Studies of temperature distribution in a cylinder heated from the outside (2) indicate that, for the temperature differences between center and surface observed near failure in these tests, the variation is intermediate between the straight line and the parabola. This is further indicated by temperature gradients in round timbers heated from the outside (3).

RESULTS OF TESTS AT ROOM TEMPERATURE

As indicated in Table II and Fig. 2, there was a nearly linear relation for each species between density and strength at room temperature. This is in general accord with results of previous tests (4). Typical stress-strain curves are given in Fig. 3.

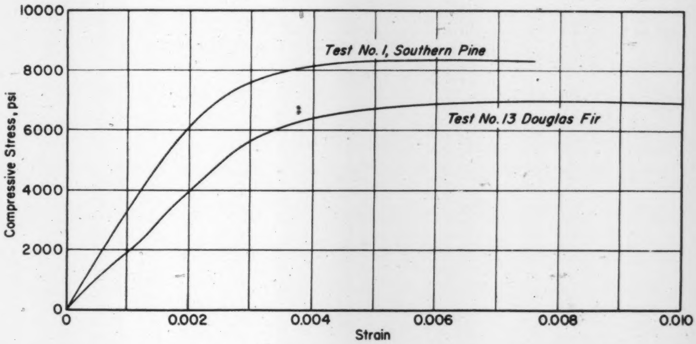


FIG. 3.—Typical Stress-Strain Curves from Compression Tests at Room Temperature.

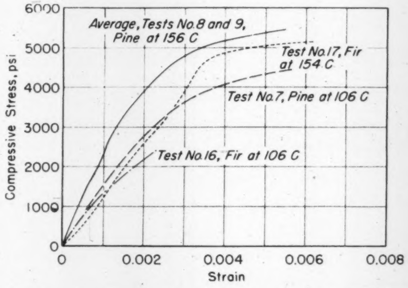


FIG. 4.—Stress-Strain Curves from Tests at Elevated Temperatures.

TESTS AT CONSTANT ELEVATED TEMPERATURES

In Table II are given results of tests in which elevated temperatures were held nearly constant while the load was increased to failure. Stress - strain curves are shown in Fig. 4, and heating time - temperature curves in Fig. 5. At 106 C, the strength was reduced to about 50 per cent of the room-temperature strength. At higher temperatures in the range 151 to 162 C, there was some regain in strength. In the only test made at still higher temperature, test No. 18 at 205 C, the strength was reduced to 62 per cent of that at room temperature, which may be compared with 83 per cent thereof for test No. 17 made at 154 C. The applicable room-temperature strength was obtained from Fig. 2 for wood of the same species and density as the specimen tested at elevated temperature.

The lower strength at 106 C, as compared with the strength at 151 to 162 C, might be attributable in part to the

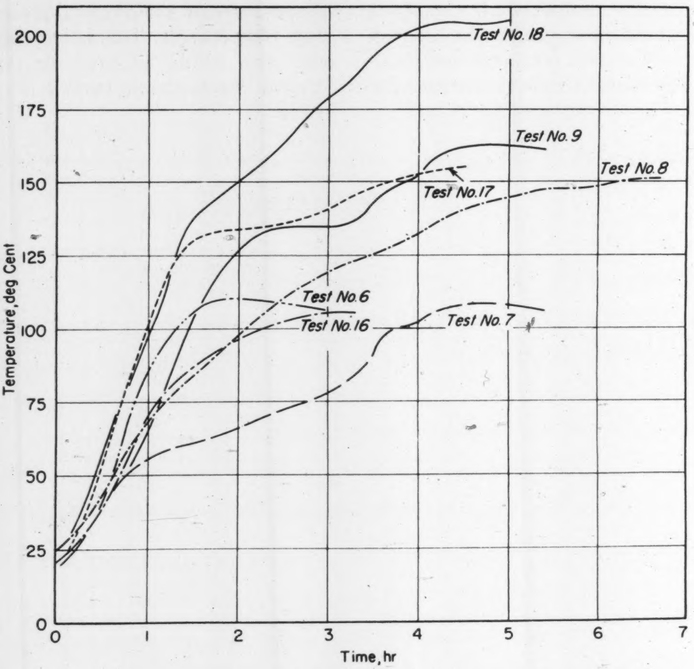


FIG. 5.—Temperatures Before and During Loading in Constant-Temperature Tests.

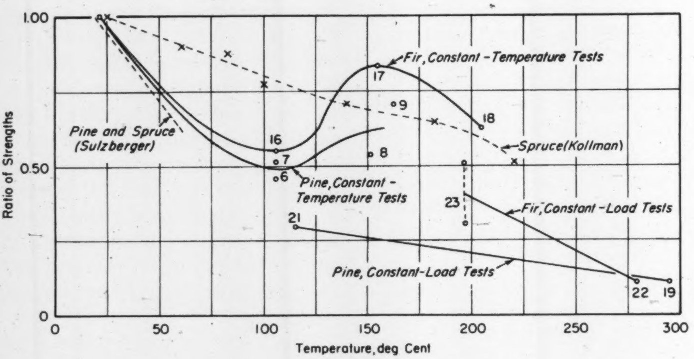


FIG. 6.—Relation of Compressive Strength at Elevated Temperatures to Compressive Strength at Room Temperature.

higher water content at the lower temperature. Also, above the higher temperature range, decrease in strength should be expected from softening of the lignin constituent of the wood. The upper two solid curves in Fig. 6 indicate the decrease in strength at elevated temperatures in tests made at constant temperatures.

Shown also in Fig. 6 are the results of two series of tests reported in the literature. As average for Sitka spruce and Australian hoop pine, Sulzberger (5) found the decrease in compressive strength between 20 and 60 C to be linear, with rate of decrease 0.93 per cent of the strength at 20 C per degree of temperature. His specimens were ½ by ½ in. by 2 in. long and had a moisture content of 15 per cent. Kollmann (6) found, with oven-dry specimens of spruce, a nearly linear decrease of strength from room temperature to 220 C. The spruce specimens, whose density was 0.43 g per cu cm, were 2 by 2 cm by 3 cm long and had an average compressive strength at room temperature of 800 kg per sq cm (11,300 psi). For tests at elevated temperatures they were heated in an oven to the desired temperature and tested in compression within 1 to 1½ min. The absence of free water with its plasticizing effect would tend to eliminate the dip in the strength curve in the region of 100 C observed in the tests here reported. The intermediate trend of the strength curves for the present tests compared with that for the tests by Sulzberger at 15 per cent moisture, and for the tests by Kollmann with oven-dry specimens should be expected on the basis of moisture content.

Later tests by Sulzberger (7) indicated a progressive decrease in the effect of temperature on strength in the range 20 to 90 C as the moisture content of the wood was decreased until, for the oven-dry condition, the results approximated those found by Kollmann.

TABLE III.—STRAINS IN CONSTANT TEMPERATURE TESTS.

Species	Tests at Elevated Temperatures				Tests at Room Temperature		Ratio of Strains, elevated: room
	Test	Temperature, deg Cent	Stress, psi	Strain, in. per in.	Test	Strain, in. per in.	
Southern pine........	No. 7	106	3440	0.00274	No. 4	0.00130	2.10
Southern pine........	No. 8	151	3880	0.00277	No. 4	0.00144	1.92
Southern pine........	No. 9	162	3940	0.00184	Nos. 1, 2	0.00122[a]	1.64
Douglas fir..........	No. 16	106	2260	0.00211	Nos. 10, 11	0.00148[a]	1.43
Douglas fir..........	No. 17	154	3740	0.00288	Nos. 10, 11	0.00239[a]	1.20
Douglas fir..........	No. 18	205	2940	0.00208	Nos. 11, 13	0.00172[a]	1.21

[a] Average for the two tests.

DEFORMATION UNDER LOAD

Table III gives the ratios of strains at elevated temperatures to those for the same loading of comparable specimens at room temperature. The ratios were highest for tests made at 106 C and decreased for tests made at higher temperatures although remaining considerably higher than for equality with strains at room temperature. The same can be seen by comparing the stress - strain curves in Fig. 4 with those in Fig. 3.

TESTS AT CONSTANT LOAD

In these tests the applied load was held constant while the temperatures were increased as shown in Fig. 7 until failure or the end of the test. Table IV gives the results in terms of the loads applied and the temperature and deformation at or near the end of the tests. The temperature required to cause failure had not been reached in test No. 20 when it was terminated at 12 hr 8 min. The lower part of Fig. 6 shows the ratios of strengths from the constant-load tests to derived room-temperature strengths. In test No. 23, with no failure under the 2000 psi applied load after 6 hr 35 min, it was increased to failure at 3320 psi. The ratios for both stresses are plotted in Fig. 6. The results indicate that, after loads are carried for longer periods as in these tests, failure occurs at lower temperatures than for comparable short-time loading as in the constant-temperature tests. After failure in test No. 19 at 294 C and in test No. 22 at 279 C, the specimens were black and in large part carbonized throughout the section.

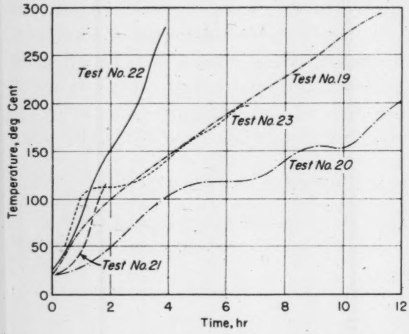


FIG. 7.—Temperatures in Constant-Load Tests.

STRAIN AND SHRINKAGE UNDER LOAD

Table IV gives the strains as determined at the last reading before failure or at the end of the heating period. While the term "strain" is used to designate the change per unit of length, an indeterminate part stems from the combined effect of expansion and shrinkage at the rising temperatures. At no time, however, did the expansion exceed the compressive deformation from application of the load, or this combined with shrinkage.

TABLE IV.—RESULTS OF TESTS AT CONSTANT LOADS.

Test	Species	Density, g per cu cm	Constant Load, psi	Part of Derived Room-Temperature Strength	Conditions at Failure		Strain, in. per in.
					Time hr:min	Temperature, deg Cent	
No. 19......	Southern pine	0.72	1030	0.11	11:12	294	0.01138
No. 20......	Southern pine	0.82	1030	0.09	12:08	208[a]	0.01682
No. 21......	Southern pine	0.70	2520	0.29	1:49	115	0.00553
No. 22......	Douglas fir	0.54	800	0.11	3:51	279	0.00945
No. 23......	Douglas fir	0.51	2000	0.30	6:35	197[b]	0.00783

[a] There was no failure at this temperature. After cooling to room temperature, the specimen was loaded to failure at 12,930 psi.

[b] There was no failure at this temperature. Load was increased to 3320 psi when failure occurred, the average temperature of the specimen being kept at 197 C. After cooling to room temperature, the specimen was loaded to failure at 3110 psi.

FIRE TESTS OF WOOD BUILDING COLUMNS

In a series of fire tests of columns (8) there were included four of longleaf Southern pine and two of Douglas fir of nominal 12 by 12 in. section with a 12-ft length exposed to fire. In the upper 1 to 1½ ft were typical bearing details consisting of a cast-iron cap and pintle or a steel plate cap. A compressive load of 920 psi was applied together with fire exposure of controlled severity until failure. The tests were accordingly of the constant-load type. For two columns with protection for cap and shaft, failure occurred at 2 hr 15 min and 1 hr 13 min, respectively, and for those without protection in the range 38½ to 50 min.

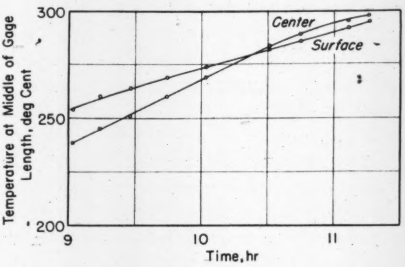


FIG. 8.—Temperature at Center and Surface Near End of Test No. 19.

The cause of failure in all tests was yielding and flow of wood at the hot metal bearings. This caused rupture of the cast-iron cap and consequent splitting of the column under the cap. The columns with the steel plate cap failed by deflecting sidewise at the cap, the wood at the bearings becoming plastic from contact with the hot cap.

To determine the fire resistance of the column as such, without limitations from bearing details, six fire tests were conducted of 12 by 12-in. timber columns (9), three of longleaf pine and three of Douglas fir. A reinforced concrete cap replaced the metal bearing details in the previous tests. With loading and fire exposure as for those with metal bearings, the columns supported the applied load for periods of 1 hr 18 min to 1 hr 52 min, the cause of failure being reduction in area of the column shaft from charring and increase in temperature of the uncharred wood. The uncharred area was from 35 to 50 per cent of the cross-section, some of the charring being subsequent to failure before the burning could be extinguished.

TABLE V.—LOSS IN WEIGHT OF SPECIMENS DURING TEST.

Test	Species	Type of Test	Duration of Heating	Temperature, deg Cent		Loss in Weight, per cent
			hr:min	Initial	At or Near End of Test	
No. 6......	Southern pine	Constant temperature	3:01	23	106	4.4
No. 7......	Southern pine	Constant temperature	5:22	24	106	4.3
No. 8......	Southern pine	Constant temperature	6:55	20	151	8.3
No. 9......	Southern pine	Constant temperature	5:25	20	162	10.2
No. 16......	Douglas fir	Constant temperature	3:20	21	106	3.5
No. 17......	Douglas fir	Constant temperature	4:54	21	154	9.0
No. 18......	Douglas fir	Constant temperature	5:26	22	205	11.2
No. 19......	Southern pine	Constant load	11:19	...	294	36.6
No. 20......	Southern pine	Constant load	12:10	21	208	17.2
No. 21......	Southern pine	Constant load	1:56	21	115	3.5
No. 22......	Douglas fir	Constant load	3:53	20	279	34.0
No. 23......	Douglas fir	Constant load	6:47	22	197	10.7

AUTOGENOUS HEATING

For tests at temperatures not over 208 C there was no evidence of self-sustained heating within the specimen, either during or after test. Fir specimen No. 18 tested at 205 C sustained a load on cooling of 83 per cent of the derived room-temperature strength (Table II). Pine specimen No. 20, on cooling after a constant-load test to 208 C, failed at a higher load than the derived strength for the room temperature condition. Fir specimen No. 23, at the end of a constant-load test to 197 C, sustained loads while hot and after cooling equal to nearly one half of the applicable room-temperature strength.

In test No. 19 with pine and No. 22 with fir at constant loads and respective end-point temperatures of 294 and 279 C, autogenous heating was indicated for the former by the time-temperature curves (Fig. 8) and for both by their condition after test. In test No. 19 the temperatures at the ends were lower than at the middle of the gage length, hence the rise at this location at the center of the cross-section above that at the surface, shown in Fig. 8, could not be attributed to heat conduction from the end bearings. The autogenous heating at these temperatures conforms with the temperatures of exothermic reaction found in connection with the destructive distillation of wood.

After cooling, both specimens were found charred throughout, the central part disintegrating more on handling than parts near the surface. The charring apparently took place in considerable part after the end of the test.

LOSS IN WEIGHT

Table V gives the loss in weight of specimens, referenced to the weight before test. The losses appear consistent with the temperatures and time of heating (Figs. 5 and 7). For tests Nos. 6 and 7, in which nearly the same loss was sustained, the time of heating with specimen temperatures near 100 C was nearly the same for the two tests. Part of the loss at higher temperatures was apparently from volatiles in the wood other than water. The losses sustained at temperatures between 275 and 300 C correspond to those found in dry distillation of wood at these temperatures.

The losses in weight of specimens tested at constant temperatures, supplemented by comparison temperature runs with similar specimens, using the same heating schedules as in the tests here reported, can be applied to evaluate the initial moisture content of the specimens for these tests. After comparison runs for tests Nos. 6, 7, and 16, with average end-point temperature of 106 C, the specimens were dried to constant weight at 102 to 103 C, the resulting losses indicating that the 3.5 to 4.4 per cent loss sustained during test was no more than about one half of the moisture content before test. Further, in comparison runs with oven-dry specimens to duplicate the temperature conditions for tests Nos. 8, 9, and 17, with average end-point temperature in the range 151 to 162 C and loss in weight during test of 8.3 to 10.2 per cent, the specimens lost 0.5 to 1.2 per cent of their weight beyond the oven-dry condition. Accordingly, in tests Nos. 8, 9, and 17, a comparable part of the loss apparently was other than water given off at or near 100 C.

Hence the losses in tests Nos. 6, 7, and 16 and those in Nos. 8, 9, and 17, all indicate initial moisture content for their specimens within the approximate range of 7 to 9 per cent, as considered in conjunction with the comparison runs. All specimens for the tests were stored under the same conditions for a sufficient length of time to enable kiln-dried material to reach moisture equilibrium, and a variation in resulting moisture content between specimens of more than 1 or 2 per cent would not be expected.

REFERENCES

(1) S. H. Ingberg and P. D. Sale, "Compressive Strength and Deformation of Structural Steel and Cast Iron Shapes at Temperatures up to 950 C," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II. p. 33 (1926).
Prentiss D. Sale, "Compression Tests of Structural Steel at Elevated Temperatures," RP741, *Journal of Research*, Nat. Bureau of Standards, Vol. 13, p. 713 (1934).

(2) H. S. Carslaw and J. C. Jaeger, "Conduction of Heat in Solids," Oxford University Press, p. 174 (1947).

(3) J. D. MacLean, "Temperatures Obtained in Timbers When the Surface Temperature is Changed After Various Periods of Heating," Fig. 6, No. R1609, U. S. Forest Products Laboratory, April 1946.

(4) L. J. Markwardt and R. E. Wilson, "Strength and Related Properties of Woods Grown in the United States," *Technical Bulletin 479*, U. S. Dept. of Agriculture, p. 36 (1935).

(5) P. H. Sulzberger, "The Effect of Temperature on the Strength Properties of Wood, Plywood, and Glued Joints," *Journal of Council for Scientific and Industrial Research*, Australia, Vol. 16, p. 263 (1943).

(6) F. Kollmann, "Die mechanischen Eigenschaften Verschiedenfeuchter Hölzer im Temperaturbereich von − 200 bis + 200 C," *Forschungsheft 403*, Verein Deutscher Ingenieure, B(11) (1940).

(7) P. H. Sulzberger, "The Effect of Temperature on the Strength of Wood, Plywood and Glued Joints," *Report ACA-46*, Aeronautical Research Consultative Committee, Australia (1953).

(8) S. H. Ingberg, H. K. Griffin, W. C. Robinson, and R. E. Wilson, "Fire Tests of Building Columns," conducted by Associated Factory Mutual Fire Insurance Companies, National Board of Fire Underwriters, and National Bureau of Standards, at Underwriters' Laboratories, Chicago, Ill., *Technologic Paper T184*, Nat. Bureau of Standards (1921).

(9) "Fire Exposure Tests of Loaded Timber Columns," *Bulletin of Research*, No. 13, Underwriters' Laboratories (1939); *Engineering News-Record*, Vol. 87, p. 972 (1921); *Contract Record and Engineering Review* (Toronto) Vol. 36, p. 1025 (1922).

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON EDUCATION IN MATERIALS

The teaching of materials in colleges of engineering has become an exceedingly critical and complicated problem and one for which there is no easy or obvious solution. Education, especially in engineering, is not static, and we are going through a period of more drastic and dramatic change than we have ever before experienced. For several years, the administrative officers of ASEE and ASTM hoped an appropriate time would come when the subject could be discussed at a jointly sponsored meeting. Each of the authors of this symposium brings to his subject a knowledge not only gained from high-level responsibilities within his own organization, but also distilled from the experience and research of others.

This symposium was developed jointly by ASTM and the American Society for Engineering Education and was held at the First and Second Sessions and a Luncheon at the Sixty-second Annual Meeting of ASTM, on June 22, 1959, in Atlantic City, N. J.

The first session was presided over by K. B. Woods, Head School of Civil Engineering, Purdue University, and President of ASTM, W. T. Alexander, President of ASEE, presided over the second session. Eric A. Walker, President, The Pennsylvania State University, delivered the address at the luncheon at which Richard T. Kropf, Belding-Heminway Co., Inc., and Past-President of ASTM, acted as Toastmaster.

The papers and the address included in the symposium are:

Introduction—K. B. Woods
Education in Engineering: a Perspective—Eric A. Walker (Education in Materials Luncheon Address)
ASTM Views on Engineering Education—F. L. LaQue
Education in Materials from an Educator's Viewpoint—W. T. Alexander
Industry's Viewpoint on Education in Materials—Melvin F. Wood
Further Challenges of the Design Engineer to the Metals Engineer and Their Bearing on the Metals Engineer's Education—Glenn B. Wârren
Nature and Properties of Materials—a Discussion of the Report of ASEE Follow-up Subcommittee on Materials—Glenn Murphy
Viewpoint of the Civil Engineer—Henry A. Lepper, Jr.
Summary of Survey on Projected Degree in Materials Engineering and Related Topics —Maurice E. Shank
Solid State Physics in Relation to Materials Science, Education, and Industry—Jacob E. Goldman

These papers, a general discussion, and the address, with the exception of the papers by Maurice E. Shank and by Jacob E. Goldman, were issued as *ASTM Special Technical Publication No. 263*, entitled "Symposium on Education in Materials."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON VISUAL AIDS FOR STANDARDIZING AND COMMUNICATING PRODUCT APPEARANCE

Committee E-12 on Appearance organized this Symposium on Visual Aids for Standardizing and Communicating Product Appearance to focus attention on the problem of product appearance specification. In today's world the rapid communication of technological advances and the astonishing and almost universal increase in technological ability have made it difficult for a product to maintain a sales advantage on the basis of engineering design alone. Appeal and sales advantage must also depend on a high level of product appearance. While some aspects of this problem belong to the industrial designer, a portion of it falls within the areas serviced by ASTM.

The papers in this symposium were presented at the Thirtieth Session of the Sixty-second Annual Meeting of the Society, held in Atlantic City, N. J., June 25, 1959. Mr. G. W. Ingle, Monsanto Chemical Co., acted as Symposium Chairman, and Mr. J. M. Hemphill, Armstrong Cork Co., presided over the session.

The papers included in the Symposium are:

Introduction—J. M. Hemphill
Status of ASTM Methods and Standards for Appearance Evaluation—I. Nimeroff
Visual Aids in the Textile Industry—J. B. Goldberg
Potential Uses of Closed Circuit Television for Product Inspection—R. Vendeland
Use of Color Photography for Product Specification and Control—J. R. Kane
Summation—G. W. Ingle

These papers, with the exception of the paper by J. R. Kane, were issued as *ASTM Special Technical Publication No. 258*, entitled "Symposium on Visual Aids for Standardizing and Communicating Product Appearance."

SUMMARY OF PROCEEDINGS OF SYMPOSIA AND SESSION ON BITUMINOUS PAVING MATERIALS

As a result of expanded highway programs to accomodate our growing traffic load and increased airport runway construction to meet the needs of increasing air traffic and the jet age, there has been an increased emphasis on better pavement construction. Highway engineers require more precise information about materials and the application of available methods of test to design and construction of bituminous paving mixtures and the development of new tests for use in the field.

ASTM Committee D-4 on Road and Paving Materials thus sponsored two Symposia, "Symposium on Methods of Test for Design of Bituminous Paving Mixtures" and "Symposium on Practical and Statistical Significance of Tests and Properties of Bituminous Binders" supplemented by "Papers on Road and Paving Materials" held and presented at four sessions during the Sixty-second Annual Meeting of the Society, June 21–26, 1959, in Atlantic City, N. J.

Mr. L. F. Rader, University of Wisconsin acted as Symposium Chairman for the Symposium on Methods of Test for Design and presided at the second session, while Mr. A. B. Cornthwaite, Department of Highway of the Commonwealth of Virginia presided at the first session, both being held on June 23.

The Symposium on Practical and Statistical Significance of Tests and Properties of Bituminous Binders was held in one session on June 24. Mr. D. F. Fink, Shell Oil Co., was the Symposium Chairman and presided at the session.

Mr. Norman W. McLeod, Imperial Oil, Ltd., was the Program Chairman for the Session on Road and Paving Materials held on June 25. Mr. C. A. Carpenter, U. S. Bureau of Public Roads, presided at the session.

The papers included in the two Symposia and the Technical Session are:

Symposium on Methods of Test for Design of Bituminous Paving Mixtures

General Factors in Design of Bituminous Paving Mixtures—L. F. Rader
Theory and Application of a Gyratory Testing Machine for Hot-Mix Bituminous Pavement—John L. McRae and Charles R. Foster
Application of the Marshall Method to Hot-Mix Design—H. L. Lehmann and Verdi Adam
Control of Bituminous Shoulder Construction for the Northern Illinois Toll Highway—Joseph J. Waddell
Triaxial Testing of Bituminous Mixtures—W. H. Goetz and J. H. Schaub
Correlation of Hveem Stabilometer and Cohesiometer Test Results and Kneading Compactor Densities with Service Records of Bituminous Pavements—Carl E. Minor
Void Requirements for Dense-Graded Bituminous Paving Mixtures—N. W. McLeod
Use of the Immersion-Compression Test in Evaluating and Designing Bituminous Paving Mixtures—Joseph F. Goode

Symposium on Practical and Statistical Significance of Tests and Properties of Bituminous Binders

Introduction—D. F. Fink
The Evolution of ASTM Tests and Specifications for Asphaltic Paving Materials—Gene Abson
Practical Significance of Tests on Asphalt Cements—Norman W. McLeod
Precision of Present ASTM Tests on Bitumens and Bituminous Materials—A. B. Brown
Fundamental Statistical Concepts in Testing—P. E. Irick
Summary—Possibilities for Extension and Improvement of ASTM Tests on Bituminous Paving Binders D. F. Fink

Papers on Road and Paving Materials

Fundamental Viscosity *versus* Saybolt Furol Viscosity for Refinery Control of Cutback Asphalt—D. F. Levy, F. E. Fassnacht, G. P. Hibler, R. D. Umbach, and D. W. Gagle
Density Changes in Asphalt Pavement Core Samples—T. C. Hein and R. J. Schmidt

These papers were issued as *ASTM Special Technical Publication No. 252*, entitled "Bituminous Paving Materials."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIA ON SOILS

The Symposium on Time Rates of Loading in Soil Testing was sponsored by Committee D-18 on Soils for Engineering Purposes and covers evaluations of soils including strain-rate effects, consolidation of clays and soils and repeated loading measurement, as well as a survey covering the literature from 1846 to 1958 on dynamic and static resistance of cohesive soils. The papers in this symposium were presented at the Sixth session of the Sixty-second Annual Meeting of the Society, held on June 22, 1959, in Atlantic City, N. J.

The Symposium on Atterberg Limits presents the history and the development of the well known Atterberg limits tests. It also covers and discusses various methods for determining the liquid limit of soils using the one-point method, the penetration tests, results obtained from various grooving tools, and the cube and standard test methods for plastic limit. The symposium was presented at the Sixteenth session of the Sixty-second Annual Meeting of the Society, held on June 23, 1959. This symposium was sponsored by Subcommittee R-3 B of Committee D-18.

Three papers on soils presented at a Session on Soils held on June 23, 1959, at the same Sixty-second Annual Meeting of the Society were added to this publication. These papers cover such problems as laboratory and field tests on cement-treated granular base-course materials, powder *versus* slurry application of lime for soil stabilization, and the determination of preconsolidation pressure of a sensitive clay.

Also included in this volume are eight papers presented at the Symposium on Soils for Engineering Purposes held on October 16, 1959 at the Third Pacific Area National Meeting of the Society in San Francisco, Calif. This symposium was sponsored by Committee D-18 on Soils for Engineering Purposes and appears in this publication as Sessions on Soils (Third Pacific Area National Meeting).

The papers cover problems related to soil density and moisture content, load and compression testing and a proposed classification system for soils.

Mr. D. M. Burmister, Columbia University, presided at the Symposium on Time Rates of Loading in Soil Testing with Mr. L. E. Gregg, L. E. Gregg and Associates, as co-chairman. The session of the Symposium on Atterberg Limits was presided over by Mr. G. F. Sowers, Law Engineering Testing Co., who also acted as chairman of the Symposium Subcommittee. The Session on Soils of the Sixty-second Annual Meeting was presided over by Mr. W. H. Goetz, Purdue University. Mr. F. J. Converse, Converse Foundation Engineering, presided over the morning session of the Symposium on Soils for Engineering Purposes and acted as Symposium Chairman; while Mr. H. de Bussieres, Curtis Tompkins, Ltd., presided over the afternoon session.

The papers included in these two Symposia and sessions on soils are:

Symposium on Time Rates of Loading in Soil Testing

Introduction—Donald Burmister
Dynamic and Static Resistance of Cohesive Soil—1846 to 1958—W. S. Housel
The Influence of Rate of Strain on Effective Stresses in Sensitive Clay—C. B. Crawford
A Method for Adjusting Strain-Rates to Obtain Pore Pressure Measurements in Triaxial Shear Tests—W. Ellis and W. G. Holtz
Apparatus for Repeated Load Tests on Soils—H. B. Seed and J. W. N. Fead
Strain-Rate Behavior of Clay and Organic Solids—D. Burmister
Automation in Consolidation Testing—R. H. Karol
Time Effects on the Consolidation of Clays—G. A. Leonards and B. K. Ramiah
The Use of Visco-Elastic Stress-Strain Laws in Soil Testing—R. L. Schiffman

Symposium on Atterberg Limits

Introduction—George F. Sowers
History and Development of the Atterberg Limits Tests—E. E. Bauer
The Use of a One-Point Liquid Limit Procedure—W. J. Eden
Ohio Adopts the One-Point Mechanical Method for Determining the Liquid Limit of Soils—J. G. Joslin and H. D. Davis
Correlation of Atterberg Limits with Geology of Deep Cores from Subsidence Areas in California—A. I. Johnson and D. A. Morris
Investigations of the Liquid Limit Test on Soils—R. F. Dawson
Liquid Limit Results from Various Types of Grooving Tools—J. E. Mitchell
Proposed Universal Standards for the Liquid Limit Tests—M. D. Morris, R. B. Ulp, and R. A. Spinna
Plastic Limit—Comparison of Cube and Standard Thread Test Methods—E. A. Abdun-Nur
Penetration Tests for Liquid Limit—G. F. Sowers, A. Vesic, and M. Grandolfi

Session on Soils (Sixty-second Annual Meeting)

Laboratory and Field Tests of Granular Soil-Cement Mixtures for Base-Courses—M. S. Abrams
Powder *versus* Slurry Application of Lime for Soil Stabilization—D. T. Davidson, G. Noguera, and J. B. Sheeler
Improved Determination of Preconsolidation Pressure of a Sensitive Clay—J. J. Hamilton and C. B. Crawford

Sessions on Soils (Third Pacific Area National Meeting)

Measurement of Excess Hydrostatic Pressures in Soils—W. G. Weber, Jr
Moisture and Strength Variation in a Thick, Uniform Clay Layer—W. G. Weber, Jr., and W. F. Kleiman
Extended Repetitive Plate Load Tests for the Evaluation and Design of Concrete Pavements—B. B. Gordon
Some Laboratory Studies of the Moisture-Density Relations of Soils—R. F. Dawson
On Determination of Dynamic Characteristics of Soils *in Situ*—R. H. Bernhard
The Effect of Temperature on Moisture Contents as Determined by Centrifuge and Tension Techniques—R. C. Prill and A. I. Johnson
The Effect of Particle Shape and Texture on the Strength of Noncohesive Aggregates—H. C. Morris
Triaxial Compression Tests on Soils Using Variable Lateral Pressure—R. H. Meese and R. W. Long
Soils Classification Triangle Based on the Unified Soil Classification System—Jack McMinn

These papers with discussion were issued as *ASTM Special Technical Publication No. 254* entitled "Soils—1959."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON IDENTIFICATION OF WATER-FORMED DEPOSITS

This Symposium on Identification of Water-Formed Deposits offers the water-treatment engineer a wide range of analytical techniques for the identification of deposits in water systems, with discussion of the advantages and disadvantages of each method.

The papers and discussions in this symposium were presented at the Thirty-Third Session of the Sixty-second Annual Meeting of the Society, held on June 25, 1959, in Atlantic City, N. J. The symposium was sponsored by Committee D-19 on Industrial Water.

Mr. R. K. Scott, Harbison Walker Refractories Co., acted as symposium chairman and presided over the session.

The papers included in the symposium are:

Introduction—R. K. Scott
Deposit Identification—First Step Toward Understanding a Water Problem—J. K. Rice
The Application of Emission Spectroscopy to the Analysis of Water-Formed Deposits —Charles H. Anderson
Identification by Instrumental Methods of Chemical Compounds in Water-Formed Deposits—C. M. Maddin and R. B. Rosene
Electron Microscopy and Electron Diffraction Studies of Oxide Films Formed on Iron in Water and Oxygen Atmospheres—Earl A. Gulbransen and Thomas P. Copan
Correlation of Elemental Analysis and Phase Identification as Viewed by a Mineralogist—J. V. Smith
Summary—R. K. Scott

These papers with discussion were issued as *ASTM Special Technical Publication No. 256* entitled "Symposium on Identification of Water-Formed Deposits."

SUMMARY OF PROCEEDINGS OF PITTSBURGH, PA. SPRING MEETING

During the 1959 Committee Week held in Pittsburgh, Pa., a special technical session was held on February 4, constituting the 1959 Spring Meeting:

SYMPOSIUM ON TESTING WINDOW ASSEMBLIES

This Symposium was sponsored by Subcommittee VIII on Windows of ASTM Committee E-6 on Methods of Testing Building Constructions. Ray B. Crepps, Purdue University, West Lafayette, Ind., was the Symposium Chairman and presided at the session.

The greatly expanded use of glass in modern architectural and engineering design, as well as the use of many kinds of material in the assemblage has increased the requirements of windows for efficient performance. Good performance now includes such factors as structural strength, adequate resistance to rain penetration and to air infiltration, and good thermal properties.

The following papers and discussion are included in the Symposium:—

Introduction—Ray B. Crepps
Evaluation of Factory-Sealed Double-Glazed Window Units—A. G. Wilson, K. R. Solvason, and E. S. Novak
Structural Testing of Large Glass Installations—C. R. Frownfelter
Norwegian Test Methods for Wind and Rain Penetration Through Windows—Sven D. Svendsen and Robert Wingen
Need for Research and Development of Test Methods for Windows—L. M. Dunn

These papers with discussion were issues as *ASTM Special Technical Publication No. 251*, entitled "Symposium on Testing Window Assemblies."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SPECTROSCOPIC EXCITATION

Excitation is one of the most important variables in emission spectroscopic analysis. The matrix effect in excitation, the effect of gaseous atmospheres on excitation, and some properties of new or modified excitation sources are covered in this symposium. Extensive discussions of these papers add greatly to their value.

The papers in this symposium were presented at the Nineteenth Session of the Sixty-second Annual Meeting of the Society, held on June 24, 1959, in Atlantic City, N. J. The symposium was sponsored by Committee E-2 on Emission Spectroscopy.

Mr. B. F. Scribner, National Bureau of Standards, acted as Chairman of the Symposium Committee and also presided over the session.

The papers included in the symposium are:

Introductory Remarks—B. F. Scribner
The Matrix Effect in Excitation—J. K. Hurwitz
Effects of Gaseous Atmospheres on Excitation—G. Andermann and J. W. Kemp
Some Properties of New or Modified Excitation Sources—M. Margoshes

These papers with discussions have been combined as *ASTM Special Technical Publication No. 259* entitled "Symposium on Spectroscopic Excitation."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON RADIOISOTOPES IN METALS ANALYSIS AND TESTING

This symposium was sponsored by Committee E-3 on Chemical Analysis of Metals in order to afford an opportunity for discussion of different applications of radioisotopes to the metals industry. It was the purpose of this symposium to provide a forum for the presentation and discussion of information in areas in which isotopes furnish unexcelled versatility and adaptability. The increasing use of analytical methods involving radioisotopes and the prospects for much greater use of such methods are dealt with in this symposium.

The papers in this symposium were presented at a special session on June 22 during the Sixty-second Annual Meeting of the Society in Atlantic City, N. J. Mr. W. W. Meinke, University of Michigan, Chairman of the Symposium Committee also presided over the sessions. The symposium was developed in two sessions: a General Session and a session on Applications in Analysis. A general panel discussion concluded the meeting.

The papers presented were:

Introduction—H. F. Beeghly

General Session

Nucleonics in Analysis—W. W. Meinke
Instrumentation and Techniques for Analysis of Radioiostopes—A. H. Emmons
Metals Analysis by Radioactivation—G. H. Morrison
Refereeing by Isotope Dilution Methods—C. Rosenblum

Applications in Analysis

Neutron Activation Analysis of Traces of Molybdenum in Tungsten—J. F. Cosgrove
Application of the (*n*, alpha) Reaction—A. H. Bushey
Experiences with Activation in the Analysis of Aluminum—J. E. Lewis
Training Industrial Personnel in Radioisotope Utilization—J. P. Danforth

These papers were issued as *ASTM Special Technical Publication No. 261* entitled "Symposium on Radioisotopes in Metals Analysis and Testing."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON HIGH VOLTAGE CABLE INSULATION

This symposium was jointly sponsored by the American Institute of Electrical Engineers, Committee on Insulated Conductors and ASTM Committee D-27 on Electrical Insulating Liquids and Gases. The objective was to bring to bear on the work and program of Committee D-27 the experience of the AIEE Insulated Conductor Committee on the problems of cable insulation and, in particular, the interrelationship of the various materials used in cables. The symposium was held on November 18, 1959, in New York City, in conjunction with meetings of both the AIEE and ASTM Committees.

Presiding at the symposium and also chairman of the symposium committee was V. R. Mulhall, Canadian General Electric Co., Peterborough, Ont., Canada.

The symposium includes the following papers:

Paper for High Voltage Cables—W. A. Delmar
Why and How of Cable Oil Tests—R. B. Blodgett
The Utility Viewpoint on Cable Oils—A. M. Gates and R. W. Gillette

These papers with general discussion are issued as *ASTM Special Technical Publication No. 253* entitled, "Symposium on High Voltage Cable Insulation."

1959 TECHNICAL PAPERS PUBLISHED IN ASTM BULLETIN

ASTM Bulletin *No. 235, January 1959*

A Method of Test for Potential Efflorescence of Masonry Mortar—P. L. Rogers, p. 31 (TP 1)

**Suitability of Lightweight Aggregate for Bituminous Plant Mix—John C. Wycoff, p. 33 (TP 3); Disc., p. 36 (TP 6)

**Extending Concrete Highway Durability and Light Reflectance with Silicones—Harold L. Cahn and Royal V. Mackey, Jr., p. 37 (TP 7); Disc., p. 41 (TP 11)

False Negative Permanent Strains Observed with Resistance Wire Strain Gages—Clarence J. Newton, p. 42 (TP 12)

Discussion of Paper on Nitric-Hydrofluoric Acid Evaluation Test for Type 316L Stainless Steel (Warren), p. 44 (TP 14)

**A Machine for Evaluation of High Temperature Alloys Under Combined Static and Dynamic Stresses—Paul E. Hawkes and C. Harold Ek, p. 46 (TP 16); Disc., p. 51 (TP 21)

Measurement of Bulk Modulus of Hydraulic Fluids—R. L. Peeler and J. Green, p. 51 (TP 21)

Accelerated Aging Tests and Life Aging Properties of Aircraft Metal Adhesives—J. P. Thomas, p. 58 (TP 28)

Resin-glass Bond Characteristics—F. J. McGarry, p. 63 (TP 33)

ASTM Bulletin *No. 236, February 1959*

The Theory of Extreme Values—J. Mandel, p. 29 (TP 39)

Should Statistical Methods Be Used to Prepare Materials Specifications—H. R. Sheppard and H. Ginsburg, p. 31 (TP 41)

Release of Alkalies by Sands and Admixture in Portland Cement Mortars—W. C. Hansen, p. 35 (TP 45)

An Instrument for the Measurement of Pore-Size Distribution by Mercury Penetration—N. M. Winslow and J. J. Shapiro, p. 39 (TP 49)

The Sucrose Extraction Method of Determining Available Calcium Oxide in Hydrated Lime—D. R. Moorehead and W. H. Taylor, p. 45 (TP 55)

Discussion of Paper on a Checking Device for Strain-Gage Indicators (Mills), p. 47 (TP 57)

A New Method for Determining the Wet Adherence of Supported Films to Various Bases—Arthur S. Diamond, p. 48 (TP 58)

A Modified Vicat Type Apparatus for Measuring the Softening Point of Thermosetting Plastics and Laminates—G. F. L. Ehlers, p. 54 (TP 64)

An Improved and Semi-Automatic Method of Conducting the Standard Hardness Test for Timber—Arno P. Schniewind, p. 57 (TP 67)

ASTM Bulletin *No. 237, April 1959*

An Improved 8-Hydroxyquinoline Method for the Determination of Magnesium Oxide in Portland Cement—H. A. Berman, p. 51 (TP 71)

Physical Changes in Setting Gypsum Plaster—B. M. O'Kelly, p. 55 (TP 75)

** Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

The Significance of Iron in Asbestos Materials Used for Electrical Insulating Purposes—P. O. Nicodemus, p. 62 (TP 82); Disc., p. 67 (TP 87)

High Precision Nondestructive Thickness Measurements of Organic Coatings—E. P. Brightwell, p. 67 (TP 87)

The Determination of Alkyl Aryl Sulfonates by Ultraviolet Absorption—R. M. Kelley, E. W. Blank, W. E. Thompson, and R. Fine, p. 70 (TP 90)

Analysis of Commercial Sodium Tripolyphosphate by Reverse Flow Ion Exchange Chromatography—R. H. Kolloff, p. 74 (TP 94)

ASTM Bulletin *No. 238, May 1959*

The Temperature Dependence of Electrical Resistivity of Laminated Thermoset Materials—Tom D. Schlabach, p. 33 (TP 101)

Shear Effects in Glass Fiber Reinforced Plastics Laminates—R. E. Chambers and F. J. McGarry, p. 38 (TP 106)

Further Development and Use of the Armstrong Sandpaper Abrasion Machine—F. M. Gavan, p. 44 (TP 112)

Fluorescent Indicator Absorption Analysis Using the Chromanalyzer—J. G. Allen and J. C. S. Wood, p. 49 (TP 117)

Determination of Ignition Loss in Portland Blast-Furnace Slag Cements—Bernard Chaiken, p. 53 (TP 121)

*Effect of Specimen Taper on Determination of Elongation in the Tension Test—Eric B. Kula and Frank R. Larson, p. 58 (TP 126)

Measurement of Surface Moisture—P. J. Sereda—Second Progress Report ASTM Committee B-3, Task Group on Measurement of Atmospheric Factors, p. 61 (TP 129)

Additional Studies with an All-Glass Multiple-Test Apparatus for Nitric Acid Testing of Stainless Steels—G. F. Tisinai and C. H. Samans, p. 64 (TP 132)

ASTM Bulletin *No. 239, July 1959*

The Interlaboratory Evaluation of Testing Methods—John Mandel and T. W Lashof, p. 53 (TP 133)

The Influence of the Direction of Loading on the Strength of Concrete Test Cubes—A. M. Neville, p. 63 (TP 143)

Prediction of Temperature Rise in Fire Testing of Walls and Partitions—R. H. Neisel, p. 66 (TP 146)

Mooney Cure Tests for Calculating Curing Times—A. E. Juve, p. 71 (TP 151)

Failure Mechanisms in Glass Fiber Reinforced Plastics— M. B. Desai and F. J. McGarry, p. 76 (TP 156)

Discussion of Paper on False Negative Permanent Strains Observed with Resistance Wire Strain Gages (Newton), p. 79 (TP 159)

ASTM Bulletin *No. 240, September 1959*

Measurement Problems in the Instrument and Laboratory Apparatus Fields—E. L. Ruh, J. J. Moran, and R. D. Thompson, p. 31 (TP 161)

Cryogenic Engineering of Hydrogen Bubble Chambers—B. W. Birmingham, D. B. Chelton, D. B. Mann, and H. P. Hernandez, p. 34 (TP 164)

Water-Retardant Qualities of External Coatings—I. Soroka, p. 40 (TP 170)

Discussion of a Paper on a Method of Test for Potential Efflorescence of Masonry Mortar (Rogers), p. 45 (TP 175)

Low-Temperature Tensile Properties of Copper and Four Bronzes—R. M. McClintock, D. A. Van Gundy, and R. H. Kropschot, p. 47 (TP 177)

---

* Presented at the Sixty-second Annual Meeting of the Society, June 21–26, 1959.

Municipal Incinerator Refractories Practice—R. B. Engdahl and J. D. Sullivan, p. 52 (TP 182)

ASTM Bulletin *No. 241, October 1959*

*An In-Place Strength Test for Low-Density Concrete—Irwin A. Benjamin and George D. Ratliff, Jr., p. 23 (TP 187); Disc., p. 25 (TP 189)

The Effect of Brick Facial Texture Upon the Initial Rate of Absorption—P. T. Hodgins, p. 27 (TP 191)

Thermal Insulation Measurements on Textiles—A Comparison of Two Methods —Paul Wing and C. J. Monego, p. 29 (TP 193)

A Powder Explosibility Tester with Continuous Feed—W. D. Box, p. 34 (TP 198)

Discussion of a Paper on Effect of Specimen Taper on Determination of Elongation in the Tension Test (Kula and Larson), p. 37 (TP 201)

High-Stress, Low-Cycle Fatigue Properties of Notched Alloy Steel Specimens—S. Yukawa and J. G. McMullin, p. 38 (TP 202)

Discussion of Paper on a Modified Vicat Type Apparatus for Measuring the Softening Point of Thermosetting Plastics and Laminates (Ehlers), p. 40 (TP 204)

*New Concrete Joint Sealant Testing Machine—R. J. Schutz and A. Van Hauter, p. 42 (TP 206)

Specimen Deflection Determination from Crosshead Travel in Three-Point Flexure Tests—C. D. Doyle and W. V. Olszewski, p. 44 (TP 208)

ASTM Bulletin *No. 242, December 1959*

Segregation in Aluminum–13 per cent Uranium Castings—D. Peckner, p. 51 (TP 211)

Design Studies of Capsules for High-Temperature Irradiations—John H. Stang, William H. Goldthwaite, and Bruce W. Dunnington, p. 54 (TP 214)

The Use of Ultrasonics in the Testing of Irradiated Fuel Elements—John M. Fouts, p. 60 (TP 220)

Control of Thermal History of Polyethylene Test Specimens—A. M. Birks and A. Rudin, p. 63 (TP 223)

Extra Durability of Paint on Low-Alloy Steels—H. R. Copson and C. P. Larrabee, p. 68 (TP 228)

Demulsibility Test for High-Viscosity Oils—A Report of Technical Committee C on Turbine Oils of ASTM Committee D-2 on Petroleum Products and Lubricants—E. W. Brennan, p. 75 (TP 235)

A Method for Determining the Demulsibility of Lubricating Oils—Paul D. Metzger, p. 77 (TP 237)

Torsion Pendulum Method for Determining Crystallinity and Void Content of Tetrafluoroethylene Resins—N. G. McCrum, p. 80 (TP 240)

1959 ANNUAL MEETING PAPER
Published in 1960

ASTM Bulletin *No. 243, January 1960*

*Control Testing for Separation of Lightweight Material from Aggregate—E. C. Higginson and G. B. Wallace, p. 60 (TP 18)

AUTHOR INDEX

VOLUME 59

A

Abdun-Nur, E. A.
Plastic Limit—Comparison of Cube and Standard Test Methods—Summary of Proceedings of *STP 254*, 1335.

Abrams, M. S.
Laboratory and Field Tests of Cement-Treated Granular Base-Course Materials—Summary of Proceedings of *STP 254*, 1335.

Abson, Gene
The Evaluation of ASTM Tests and Specifications for Asphaltic Paving Materials—Summary of Proceedings of *STP 252*, 1333.

Achter, M. R.
See Danek, G. J., Jr. and Achter, M. R.

Adam, L. H. and Dougherty, Marie
Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys. Report of Committee B-7 (appendix III), 289.

Adams, A. B.
See Prior, M. R. and Adams, A. B.

Adams, Charles
Ultrasonic Standards for the Evaluation of Missile Materials and Components—Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 278*).

Adams, R. F.
See Tuthill, L. H. Adams, R. F., and Hemme, J. M., Jr.

Adler, Isidore
Application of X-ray Spectroscopy to Unsolved Problems in Geochemistry—Symposium on Spectroscopy (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 269*).

Albertson, C. P.
An Ultrasonic Method to Detect, Count and Measure Particle Size of Contamination in Fluids—Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 278*).

Alden, R. C.
Research Comes First, ACR notes. Published in ASTM BULLETIN, No. 235, Jan., 25.

Alexander, W. T.
Education in Materials from an Educator's Viewpoint—Summary of Proceedings of *STP 263*, 1330.

Alger, J. V. and Skupien, L. M.
Neutron Radiation Embrittlement at 500 and 650 F of Reactor Pressure Vessel Steels—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 276*).

Allen, J. G. and Wood, J. C. S.
Fluorescent Indicator Absorption Analysis Using the Chromanalyzer. Published in ASTM BULLETIN, No. 238, May, 49 (TP 117).

Allred, H. M.
Application of Electron Microscopy in the Petroleum Industry—Summary of Proceedings of *STP 257*, 1074.

Andermann, G. and Kemp, J. W.
Effects of Gaseous Atmospheres on Excitation—Summary of Proceedings of *STP 259*, 1338.

Anderson, Charles H.
The Application of Emission Spectroscopy to the Analysis of Water-Formed Deposits—Summary of Proceedings of *STP 256*, 1336.

Anderson, R. M.
See Harris, J. C. and Anderson, R. M.

Armstrong, Ellis L.
Research Today . . . for Better Roads Tomorrow—Condensation of an address, 62nd ASTM Annual Meeting. Published in ASTM BULLETIN No. 241, Oct., 14.

Austin, Arthur L. and Steidel, Robert F., Jr.
The Tensile Properties of Some Engineering Materials at High Rates of Strain, 1292 (*R*). Disc., 1307.

B

Bade, W. M.
See John, R. R. and Bade, W. M.

Baechler, R. H.
Determining Preservative Retention in Piling by the Assay of Borings—Symposium on Treated Wood for Marine Use—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 275*).

Baird, R. B.
See Lipsitt, H. A., Forbes, F. W., and Baird, R. B.

Baird, R. B., Forbes, F. W., and Lipsitt, H. A.
Tensile and Fatigue Properties of Laminate Sheet Structures, 755 (*R*).

Ballantine, D. S.
The Significance to ASTM of Post-Irradiation Effects in Irradiated Materials—Symposium on Post-Irradiation Effects in Polymers—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 276*).

Bare, D. W.
See Carlson, O. N., Bare, D. W., Gibson, E. D., and Schmidt, F. A.

Barker, F. B.
Determination of Radioactive Materials in Water—Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 273*).

Barnes, S. B.
The Testing of Diaphragms to Determine Adequacy in Resisting Horizontal Forces—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 275*).

Barr, R. Q.
See Semchyshen, M. and Barr, R. Q.

Barron, L. A.
Discussion. Published in ASTM BULLETIN, No. 241, Oct., 26 (TP 190).

Barrows, R. F.
See Newell, D. M. Kerlin, E. E., Bauerlein, R. R., and Barrows, R. F.

Bauer, E. E.
History and Development of the Atterberg Limits Tests—Summary of Proceedings of *STP 254*, 1335.

Bauerlein, R. R.
See Newell, D. M. Kerlin, E. E., Bauerlein, R. R., and Barrows, R. F.

Beaufait, L. J.
See Manov, G. G. and Beaufait, L. J.

Beaver, W. W.
See Hurd, J. N., O'Rourke, R. G., and Beaver, W. W.

Beaver, W. W., O'Rourke, R. G., and Hurd, J. N.
Effect of Purity and Manufacturing Variables on the Elevated Temperature Properties of Beryllium—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 272*).

Beeghly, H. F.
Introduction—Summary of Proceedings of *STP 261*, 1339.

Begley, R. T.
See Manjoine, M. J., France, L. L., and Begley, R. T.

Begley, R. T. and France, L. L.
The Effect of Oxygen and Nitrogen on the Workability and Mechanical Properties of Columbium (Niobium)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 272*).

Benjamin, I. A.
Discussion, 1229.

Benjamin, Irwin A. and Ratliff, George D., Jr.
An In-Place Strength Test for Low-Density Concrete. Published in ASTM BULLETIN No. 241, Oct., 23 (TP 187). Disc., 25 (TP 189).

Berman, H. A.
An Improved 8-Hydroxyquinoline Method for the Determination of Magnesium Oxide in Portland Cement. Published in ASTM BULLETIN No. 237, April, 51 (TP 71).

Bernhard, R. H.
On Determination of Dynamic Characteristics of Soils *in situ*—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 54—Summary of Proceedings of *STP 254*, 1335.

Best, George E., Hecker, J. G., Jr., McGrew, J. W., and VandenBerg, R. V.,
Chromic Acid Anodizing Characteristics of Wrought Aluminum Alloys. Report of Committee B-7 (appendix II), 277 (*R*).

Bieber, C. G. and Decker, R. F.
Effect of Heat Treatment on a cast Nickel Alloy—Summary of Proceedings of *STP 262*, 1072.

Biggs, R. A.
Some Experience and Aspects of Structural Sandwich Applications in Building Construction—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 270*).

Binder, D.
See Parkinson, W. W. and Binder, D.

Biondi, F. J.
See Standing, S. A. and Biondi, F. J.

Birks, A. M. and Rudin, A.
Control of Thermal History of Polyethylene Test Specimens. Published in ASTM BULLETIN No. 242, Dec., 63 (TP 223).

Birks, L. S.
Basic Practices in X-ray Fluorescence—Symposium on Spectroscopy (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 269*).
Discussion. Published in ASTM BULLETIN, No. 235, Jan., 51 (TP 21).

Birks, L. S. and Seebold, R. E.
Composition δ'^2 Phase in High-Temperature Alloys from Electron Microprobe Measurements—Summary of Proceedings of *STP 262*, 1072.

Birmingham, B. W., Chelton, D. B., Mann, D. B., and Hernandez, H. P.
Cryogenic Engineering of Hydrogen Bubble Chambers. Published in ASTM BULLETIN No. 240, Sept., 34 (TP 164) (*R*).

Bjorksten, Johan
See Lappala, R. P., Yaeger, L. L., and Bjorksten, Johan

Blaine, R. L.
See Crandall, J. R. and Blaine, R. L.

Blank, E. W.
See Kelley, R. M. Blank, E. W., Thompson, W. E., and Fine, R.

Bloch, Peter
Ultrasonic Gaging of Missile Components During Manufacture—Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 278*).

Blodgett, R. B.
Why and How of Cable Oil Tests. Summary of Proceedings of *STP 253*, 1340.

Bloom, F. K.
Classification of Materials and Stress Conditions. Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels (*published in STP 264*).
Steps Taken to Overcome Cracking. Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels (*published in STP 264*).

Bly, D. D.
See Mellon, M. G. and Bly, D. D.

Bly, J. H. and Burrill, E. A.
High-Energy Radiography in the 6- to 30-Mev Range—Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 278*).

Bolt, R. O.
See Hall, K. L., Klaver, R. F., Carroll, J. G., and Bolt, R. O.

Borachia, P.
See Marin, J., Borachia, P., and Rimrott, U. A.

Boscia, T. D.
See Moore, R. J., Boscia, T. D., and Lundeen, R. W.

Box, W. D.
A Powder Explosibility Tester with Continuous Feed. Published in ASTM BULLETIN, No. 241, Oct., 34 (TP 198) (*R*).

Brady, R. R.
AISI Type 304L Stainless Steel with Improved Strength, 774. Disc., 785.
See Brickner, K. G. and Brady, R. R.

Brattain, R. R.
Report on Three European Spectroscopy Meetings Attended Early in 1959—Symposium on Spectroscopy (Session VI)—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 269*).

Brennan, E. W.
Demulsibility of High Viscosity Oils—Report of Technical Committee C on Turbine Oils of ASTM Committee D-2 on Petroleum Products and Lubricants. Published in ASTM BULLETIN No. 242, Dec., 75 (TP 235).

Brenner, Abner
History of the Electroless Plating Process—Summary of Proceedings of *STP 265*, 1073.

Brickner, K. G. and Brady, R. R.
Discussion, 802.

Brightwell, E. P.
High Precision Nondestructive Thickness Measurements of Organic Coatings. Published in ASTM BULLETIN No. 237, April 67 (TP 87).

Brinn, J.
See Martin, D. E. and Brinn, J.

Brodrick, Ronald F.
Discussion, 721.

Brooks, W. B. and Holmberg, M. E.
Classification of Corrosion Conditions. Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels (*published in STP 264*).

Brown, A. B.
Precision of Present ASTM Tests on Bitumens and Bituminous Materials—Summary of Proceedings of *STP 252*, 1333.

Brown, A. B. and Sparks, S. W.
This is your Roofing Asphalt—Composition and Rheology—Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 280*).

Brown, C. T.
See Howes, J. E., Jr., Brown, C. T. Sunderman, D. N., Pobereskin, M., and Schall, P.

Brown, R. E.
See Schwendiman, L. C., Brown, R. E., Honstead, J. F., Linderoth, C. E., and Pearce, D. W.

Brown, W. F., Jr.
See Espey, G. B., Jones, M. H., and Brown, W. F., Jr.
See Epsey, G. B., Repko, A. J., and Brown, W. F., Jr.
See Sachs, G., Sell, R., and Brown, W. F., Jr.

Brunauer, Stephen, Copeland, L. E., Kantro, D. L., Weise, C. H., and Schultz, Edith G.
Quantitative Determination of the Four Major Phases in Portland Cements by X-ray Analysis, 1091.

Brunberg, W. C.
Standardization's Role in the Purchasing Function—Symposium on Standards—Summary of Proceedings of Third Pacific Area Meeting, 55 (*publication pending*).

Buell, B. E.
Special Problems in the Determination of Tetraethyllead in Gasoline by Flame Photometry—Symposium on Spectroscopy (Session III)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 269*).

Buhler, R. D.
Arc jet Kinetics Related to Ablation Test Validity—Symposium on Thermal Ablation (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 279*).

Burghoff, H. L., Kuzmenko, P., and Nole, V. F.
Discussion, 1046.

Burmister, D.
Introduction—Summary of Proceedings of *STP 254*, 1335.
Strain-Rate Behavior of Clay and Organic Solids—Summary of Proceedings of *STP 254*, 1335.

Burrill, E. A.
See Bly, J. H. and Burrill, E. A.

Bushey, A. H.
Application of the (n, alpha) Reaction—Summary of Proceedings of *STP 261*, 1339.

Butterfield, W. L. and Haney, F. H.
Asphaltic Grouts, Mastics, and Cold Applied Cements—Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 280*).

C

Cahn, Horold L. and Mackey, Royal V., Jr.
Extending Concrete Highway Durability and Light Reflectance with Silicones. Published in ASTM BULLETIN No. 235, Jan., 37 (TP 7). Disc., 41 (TP 11).

Caldarella, G. J.
See Degering, E. F., Caldarella, G. J. and Mancini, M. A.

Calkins, V. P.
Introduction—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 276*).

Cameron, Eugene N.
The Study of Opaque Minerals in Reflected Light—Summary of Proceedings of *STP 257*, 1074.

Candela, Felix
The Moment of Truth. Adventure in Full Scale Shell Tests—Cement and Concrete Industry Luncheon—Summary of Proceedings of Third Pacific Area Meeting, 38 (*not to be published*).

Carlson, O. N., Bare, D. W., Gibson, E. D., and Schmidt, F. A.
Survey of the Mechanical Properties of Yttrium and Yttrium Alloys—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 272*).

Carroll, J. G.
See Hall, K. L., Klaver, R. F., Carroll, J. G., and Bolt, R. O.

Carwile, N. L.
See Geil, G. W. and Carwile, N. L.

Caton, R. D.
See Freund, H. and Caton, R. D.

Caugherty, Betty, Oatman, H. T., and Reen, O. W.
A Cooperative Study of the Hardness Testing of Cemented Carbides, 1261. Disc., 1275.

Cestaro, J. P.
See Dunn, E. J., Cestaro, J. P., and Von Weisenstein, W.

Chaiken, Bernard
Determination of Ignition Loss in Portland Blast-Furnace Slag Cements. Published in ASTM BULLETIN, No. 238, May, 53 (TP 121).

Chamberlain, M. D.
See Fair, W. F., Jr. and Chamberlain, M. D.

Chambers, R. E. and McGarry, F. J.
Shear Effects in Glass Fiber Reinforced Plastics Laminates. Published in ASTM BULLETIN, No. 238, May 38 (TP 106).

Chartz, J. A.
Our Products *versus* Your Tests—Electronics Industry Luncheon Address—Summary of Proceedings of Third Pacific Area Meeting, 37 (*not to be published*).

Chelton, D. B.
See Birmingham, B. W., Chelton, D. B., Mann, D. B., and Hernandez, H. P.

Chisholm, R. A.
Discussion. Published in ASTM BULLETIN, No. 235, Jan., 36 (TP 6).

Clark, Frank E.
Old Wine in New Bottles—A New Look at the Substance Water—Water Industry Luncheon Address—Summary of Proceedings of Third Pacific Area Meeting, 38. Published in ASTM BULLETIN, No. 243, Jan., 1960, p. 17.

Coffin, L. F., Jr.
See Tavernelli, J. F. and Coffin, L. F., Jr.

Coggeshall, N. D. and Hubis, W.
The Combination of Methods in the Analysis of Complex Hydrocarbon Systems—Symposium on Spectroscopy (Session VI)—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 269*).

Cohen, Bennie
Test Methods—Summary of Proceedings of *STP 265*, 1073.

Coil, J. A.
See King, H. F. and Coil, J. A.

Comer, Joseph J.
The Electron Microscope in the Study of Minerals and Ceramics—Summary of Proceedings of *STP 257*, 1074.

Connor, C. C.
Discussion, 1088.

Conrow, A. D.
An Approach to the Problem of Evaluation of Plasticity and Workability of Pastes and Mortars, 1075. Disc., 1086.
Discussion, 1202.

Conway, John
Spectroscopy of Radioactive Materials—Symposium on Spectroscopy (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 269*).

Copan, Thomas P.
See Gulbransen, Earl A. and Copan, Thomas P.

Copeland, L. E.
See Brunauer, Stephen, Copeland, L. E. Kantro, D. L., Weise, C. H., and Schultz, Edith G.

Copeland, R. E.
Structural Bond of Mortar and Grout to Concrete Block—Symposium on Masonry Materials—Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*).

Copson, H. R. and Larrabee, C. P.
"Extra Durability of Paint on Low-Alloy Steels" Published in ASTM BULLETIN, No. 242, Dec., 68 (TP 228) (*R*).

Cornet, I.
See Gallacio, A. and Cornet, I.

Corten, Herbert T.
Discussion, 675, 732, 748, 914, 983, 1013.

Cosgrove, J. F.
Neutron Activation Analysis of Traces of of Molybdenum in Tungsten—Summary of Proceedings of *STP 261*, 1339.

Cousineau, R. D.
Testing of Large Diaphragms—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 275*).

Crandall, J. R. and Blaine, R. L.
Statistical Evaluation of Interlaboratory Cement Tests, 1129.

Crawford, C. B.
Influence of Rate of Strain on Effective Stresses in Sensitive Clay—Summary of Proceedings of *STP 254*, 1335.
See Hamilton, J. J. and Crawford, C. B.

Crepps, Ray B.
Introduction—Summary of Proceedings of *STP 251*, 1337.

Cresap, Mark W., Jr.
Does Management Get Full Value from Purchasing? Published in ASTM BULLETIN, No. 242, Dec., 35.

Criscuolo, E. L., Polansky, D., Halloway, J., and Dyer, C. H.
Radiography of Large Solid Propellant Rocket Motors—Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 278*).

Crooks, Roland D.
Discussion, Published in ASTM BULLETIN, No. 243, Jan., 1960, 54 (TP 12).

Cross, H. C.
Discussion, 983.

Crossmon, Germain
Chemical Microscopy in the Optical Industry—Summary of Proceedings of *STP 257*, 1074.

Cummings, Ross
Use of Modified McMichael Viscometer for Consistency of Printing Inks and Ink Vehicles—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).
Use of Stromer Viscometer for Testing Rotogravure and Flexographic Inks—Session on Paints and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).

Cummins, E. W.
Discussion, 1214.

Curley, E. A.
Quantitative Light Element Analysis, Fe to Mg, for Portland Cement by X-ray Spectrography—Symposium on Spectroscopy (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 269*).

Currin, C. G.
Post-Irradiation Dielectric Properties of Silicones—Symposium on Post-Irradiation Effects in Polymers—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 276*).

D

Dana, A. W., Jr. and Friend, W. Z.
General Information. Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels (*published in STP 264*).
Survey of Current Research Activities. Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels (*published in STP 264*).

Danald, G. E.
Standardization of Terminology for Gamma and Electron Beam Radiation Sources—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 276*).

Danek, G. J., Jr. and Achter, M. R.
A Servomechanism for Vacuum and Controlled-Environment Fatigue Testing. Published in ASTM BULLETIN, No. 239, July 80 (TP 160). (Technical Note).

Danforth, J. P.
Training Industrial Personnel in Radioisotope Utilization—Summary of Proceedings of *STP 261*, 1339.

D'Appolonia, E.
See Romualdi, J. P. and D'Appolonia, E.

Davidson, D. T., Noguera, G., and Sheeler, J. B.
Powder *versus* Slurry Application of Lime for Soil Stabilization—Summary of Proceedings of *STP 254*, 1335.

Davis, H. D.
See Joslin, J. G. and Davis, H. D.

Davis, Raymond E.
Discussion, 1202.

Dawson, R. F.
Investigations of the Liquid Limit Test on Soils—Summary of Proceedings of *STP 254*, 1335.
Some Laboratory Studies of the Moisture-Density Relations of Soils—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 52—Summary of Proceedings of *STP 254*, 1335.

Decker, R. F.
See Bieber, C. G. and Decker, R. F.

Degering, E. F., Caldarella, G. J., and Mancini, M. A.
Post-Irradiation Effects: Monomers and Polymers—Symposium on Post-Irradiation Effects in Polymers—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 276*)

deGruy, Ines V.
See Rollins, Mary L., deGruy, Ines V., Tripp, Verne W., and Moore, Anna T.

Delmar, W. A.
Paper for High Voltage Cables. Summary of Proceedings of *STP 253*, 1340.

Denhard, E. E.
Equipment, Mode, Location of Fractures, and Associated Conditions. Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels (*published in STP 264*).

Desai, M. B. and McGarry, F. J.
Failure Mechanisms in Glass Fiber Reinforced Plastics. Published in ASTM BULLETIN, No. 239, July, 76 (TP 156).

Diamond, Arthur S.
A New Method for Determining the Wet Adherence of Supported Films to Various Bases. Published in ASTM BULLETIN, No. 236, Feb., 48 (TP 58) (*R*).

Dickinson, Dale
Discussion, 1013.

Dill, F. H.
Discussion. Published in ASTM BULLETIN, No. 241, Oct., 37 (TP 201).

Dillon, C. P.
Discussion. Published in ASTM BULLETIN, No. 235, Jan., 44 (TP 14).

Dise, J. R.
The Cement Reference Laboratory (1929–1959). Report of Committee C-1 (appendix II), 369.

Dockweiler, E. V. and Stover, H. E.
A proposed Standard Rating System for the Evaluation of Wood Piles in Sea Water—Symposium on Treated Wood for Marine Use—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 275*).

Dolan, T. J.
Discussion, 659.

Dolan, Thomas J.
Basic Research in Fatigue of Metals. Published in ASTM BULLETIN, No. 240, Sept., 24 (*R*).

Dolch, W. L.
Studies of Limestone Aggregates by Fluid-Flow Methods, 1204 (*R*). Disc., 1214.

Donaldson, W.
See Herzog, S. H. and Donaldson, W.

Donegan, J. W.
See Pittman, C. U. and Donegan, J. W.

Dougherty, Marie
See Adam, L. H. and Dougherty, Marie

Douglass, R. W.
See Holden, F. C., Douglass, R. W., and Jaffee, R. I.

Dowell, A. M.
See Hribar, V. F. and Dowell, A. M.

Doyle, C. D. and Olszewski, W. V.
Specimen Deflection Determination from Crosshead Travel in Three-Point Flexure Text. Published in ASTM BULLETIN, No. 241, Oct., 44 (TP 208).

Dranichak, Michael
Discussion. Published in ASTM BULLETIN, No. 235, Jan., 41 (TP 11).

Drisko, R. W. and Hochman, Harry
The Use of Column Chromatography in the Analysis of Creosote—Symposium on Treated Wood for Marine Use—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 275*).

Dulis, E. J.
Discussion, 833.
See Kasak, A., Hsiao, C. M., and Dulis, E. J.

Dunn, E. J., Jr., Cestaro, J. P., and Von Weisenstein, W.
Suggested Method for Spectrochemical Analysis of Antimony by the Point-to-Plane Spark Technique. Report of Committee E-2 (appendix I), 573.

Dunn, L. M.
Need for Research and Development of Test Methods for Windows—Summary of Proceedings of *STP 251*, 1337.

Dunnington, Bruce W.
See Stang, John H., Goldthwaite, William H., and Dunnington, Bruce W.

Dyer, C. H.
See Criscuolo, E. L., Polansky, D., Halloway, J., and Dyer, C. H.

E

Ebeling, Walter
Termite Control in California Residential Construction—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 275*).

Eberly, W. S.
Magnetic Materials and Testing (Committee A-6 on Magnetic Properties)—Symposium on Materials in the Electronics Industry—Summary of Proceedings of Third Pacific Area Meeting, 43 (*not to be published*).

Eby, R. E.
Deflection Limitations as a Factor in Structural Timber Design—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 46, (*to be published in STP 275*).

Edberg, E. A.
Effect on Climatic Variations on the Performance of Polysterene Foam Core Sandwich Panels in Roofs and Walls—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 270*).

Eden, W. J.
The Use of One-Point Liquid Procedure—Summary of Proceedings of *STP 254*, 1335.

Edgerton, N. W.
Discussion. Published in ASTM BULLETIN No. 237, April, 67 (TP 87).

Ehlers, G. F. L.
A Modified Vicat Type Apparatus for Measuring the Softening Point of Thermosetting Plastics and Laminates. Published in ASTM BULLETIN No. 236, Feb. 54 (TP 64). Disc., No. 241, Oct., 40 (TP 204).

Ek, C. Harold
See Hawkes, Paul E. and Ek, C. Harold

Ekse, Martin and Morris, H. C.
A Test for Production of Plastic Fines in the Process of Degradation of Mineral Aggregates—Symposium on Road and Paving Materials (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 277*).

Ellis, W. and Holtz, W. G.
A Method for Adjusting Strain-Rates to Obtain Pore Pressure Measurements in Triaxial Shear Tests—Summary of Proceedings of *STP 254*, 1335.

Emmons, A. H.
Instrumentation and Techniques for Analysis with Radioisotopes—Summary of Proceedings of *STP 261*, 1339.

Endicott, H. S.
Current Activities of Committee D-9—Symposium on Electrical Insulating Materials—Summary of Proceedings of Third Pacific Area Meeting, 41 (*publication pending*).
Insulation and Printed Wiring (Committee D-9 on Electrical Insulating Materials)—Symposium on Materials in the Electronics Industry—Summary of Proceedings of Third Pacific Area Meeting, 43 (*not to be published*).

Engdahl, R. B. and Sullivan, J. D.
Municipal Incinerator Refractories Practice. Published in ASTM Bulletin, No. 240, Sept., 52 (TP 182).

Engell, H. J.
Discussion, 1291.

Enomoto, N.
A Method for Determining the Fatigue Limit of Metals by Means of Stepwise Load Increase Test, 711 (*R*). Disc., 721.

Epstein, G. and Gary, W. W., Jr.
Permeability of Materials Under High Pressures and at Various Temperatures—Symposium on Reinforced Plastics—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 279*).

Erickson, E. C. O. and Wood, L. W.
Strength and Related Properties of Wood-Poles—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 275*).

Erickson, P. W.
See Silver, I., Erickson, P. W., and Perry, H. A.

Espey, G. B., Jones, M. H., and Brown, W. F., Jr.
Sharp Edge Notch Tensile Strength of Several High-Strength Steel Sheet Alloys, 837 (*R*). Disc., 872.

Espey, G. B., Repko, A. J., and Brown, W. F., Jr.
Effect of Cold Rolling and Stress Relief on the Sharp Edge Notch Tensile Characteristics of Austenitic Stainless Steel Sheet Alloys, 816 (*R*). Disc., 833.

Ewell, Raymond
Soviet Russia Poses a New Industrial Threat. Published in ASTM Bulletin No. 239, July, 43.

Eysenbach, G. E. and Gouza, J. J.
Improvements in ASTM Flammability Test Method. Published in ASTM Bulletin, No. 242, 62 (TP 222).

F

Fair, W. F., Jr. and Chamberlain, M. D.
Coal Tar Flat Roof Pitch—Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 280*).

Fassnacht, F. E.
See Levy, D. F., Fassnacht, F. E., Hibler, G. P., Umbach, R. D., and Gagle, D. W.

Fead, J. W. N.
See Seed, H. B. and Fead, J. W. N.

Feldstein, M.
The Colorimetric Determination of Formaldehyde and Methanol from Combustion Sources—Symposium on Air Pollution Control—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 281*).

Felgar, R. P.
Fatigue Tests on Three Cast Irons at Elevated Temperatures, 767.

Feltner, C. E.
Discussion, 673, 686, 731, 1032.

Fenn, R. W.
Short-Time Compression-Creep Testing of Sheet Utilizing High-Speed Heating—Summary of Proceedings of Third Pacific Area Meeting, 49 (*publication pending*).

Fine, R.
See Kelley, R. M., Blank, E. W., Thompson, W. E., and Fine, R.

Fink, D. F.
Introduction—Summary of Proceedings of *STP 252*, 1333.
Summary—Possibilities for Extension and Improvement of ASTM Tests on Bituminous Paving Binders—Summary of Proceedings of *STP 252*, 1333.

Finnerty, J. J.
Utilization of Cooling Towers in Conservation and Pollution Control Programs—Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 273*).

Fischer, H. C.
See MacPherson, D. R. and Fischer, H. C.

Fisher, John C.
The Role of Dislocations in Plastic Deformation (of Metals). Published in ASTM BULLETIN, No. 237, April, 7; No. 241, Oct., 6 (synopsis). (Gillett Lecture) (*published as separate pamphlet.*)

Fisher, R. M. and Szirmae, A.
Electron Microscope Observations of Dislocations in Thin Metal Foils—Summary of Proceedings of *STP 262*, 1072.

Flinn, R. A.
See Trojan, Paul K., Flinn, R. A., and Reese, D. J.

Forbes, F. W.
See Baird, R. B., Forbes, F. W., and Lipsitt, H. A.
See Lipsitt, H. A., Forbes, F. W., and Baird, R. B.

Forbes, F. W. and Shilton, R.
Band Nomenclature for the Ultraviolet Spectra of Conjugated Organic Compounds —Symposium on Spectroscopy (Session IV) —Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 269*).

Foster, Bruce
Introduction and Summary—Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 45, 47 (*to be published in STP 266*).

Foster, Charles R.
See McRae, John L. and Foster, Charles R.

Foster, G. B.
Radioisotope Methods of Testing Uniformity of Coated Fabrics—Symposium on Applied Radiation and Radioisotope Test Methods —Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 268*).

Fouts, J. M.
The Use of Ultrasonics in the Testing of Irradiated Fuel Elements. Published in ASTM BULLETIN, No. 242, Dec., 60 (TP 220).

France, L. L.
See Begley, R. T. and France, L. L.
See Manjoine, M. J., France, L. L., and Begley, R. T.

Francis, C. E. and Sipple, H. E.
Emulsion Type Fire Resistant Industrial Hydraulic Fluids—Symposium on Hydraulic Fluids (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 267*).

Freund, H. and Caton, R. D.
Coulometric Determination of Tin with Electrically Generated Iodine: Application to Analysis of Zircaloy—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 272*).

Fricke, William, G., Jr.
Discussion, 750.

Friedel, R. A.
Ultraviolet Spectroscopy: Aromaticity; Absorption Errors—Symposium on Spectroscopy (Session VI)—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 269*).

Friend, W. Z.
See Dana, A. W., Jr. and Friend, W. Z.

Frownfelter, C. R.
Structural Testing of Large Glass Installations—Summary of Proceedings of *STP 251*, 1337.

Fry, D. L.
Communications in Spectrochemical Analysis —Symposium on Spectroscopy (Session I)—Summary of Third Pacific Area Meeting, 40 (*to be published in STP 269*).

Furby, N. W. and Hanly, F. J.
Development of Improved Insulating Oils—Symposium on Electrical Insulating Materials—Summary of Proceedings of Third Pacific Area Meeting, 41 (*publication pending*).

G

Gagle, D. W.
See Levy, D. F., Fassnacht, F. E., Hibler, G. P., Umbach, R. D., and Gagle, D. W.

Gallaccio, A. and Cornet, I.
Marine Atmosphere Exposure Coupled to Form Galvanic Cells Involving Magnesium (*issued as STP 255*).

Gambrill, C. M.
Expanding Horizons in Analytical Chemistry. Published in ASTM BULLETIN No. 239, July, 45.

Garofalo, F.
Creep-Rupture Behavior of Notched and Unnotched Specimens of Types 304, 316, and 321 Austenitic Stainless Steels, 957 (*R*).
Effect of Environment on Creep and Creep-Rupture Behavior of Several Steels at Temperatures of 1000 to 1200 F, 973 (*R*). Disc., 983.
Discussion, 1071.

Gary, W. W., Jr.
See Epsteien, G. and Gary, W. W., Jr.

Gates, A. M. and Gillette, R. W.
The Utility Viewpoint on Cable Oils. Summary of Proceedings of *STP 253*, 1340.

Gates, L. E. and Lent, W. E.
Preliminary Evaluation of Ceramic Adhesives for Stainless Steel—Symposium on Adhesion and Adhesives—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 271*).

Gavan, F. M.
Further Development and Use of the Armstrong Sandpaper Abrasion Machine. Published in ASTM BULLETIN No. 238, May, 44 (TP 112).

Gazzara, C. P.
See Klier, E. P. and Gazzara, C. P.

Geil, G. W.
Discussion, 1029.

Geil, G. W. and Carwile, N. L.
Effect of Notch Geometry on Tensile Properties of Annealed Titanium at 100, 25, −78, and −196 C, 985 (*R*). Disc., 1013.

Gerard, George
See Tramposch, Herbert, Papirno, Ralph, and Gerard, George.

Gibson, E. D.
See Carlson, O. N., Bare, D. W., Gibson, E. D., and Schmidt, F. A.

Gilbert, P. T., Jr.
Analytical Flame Photometry: New Developments—Symposium on Spectroscopy (Session III)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 269*).

Gilbraith, Allen C.
A New Steel for Aircraft and Missile Honeycombs. Published in ASTM BULLETIN No. 240, Sept., 51 (TP 181). (Technical Note.)

Gillette, R. W.
See Gates, A. M. and Gillette, R. W.

Ginsburg, H.
See Sheppard, H. R. and Ginsburg, H.

Glaze, R. A.
Need for Special Engineering Education in the Field of Structural Timber Design—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 275*).

Gloger, W. A.
Discussion of the Work of Committee D-1—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).

Goetz, W. H.
Discussion. Published in ASTM BULLETIN, No. 235, Jan., 36 (TP 6).

Goetz, W. H. and Schaub, J. H.
Triaxial Testing of Bituminous Mixtures—Summary of Proceedings of *STP 252*, 1332.

Gohn, G. R.
Discussion, 709.

Goldberg, J. B.
Visual Aids in the Textile Industry—Summary of Proceedings of *STP 258*, 1331.

Goldman, Jacob E.
Solid State Physics in Relation to Materials Science, Education and Industry—Summary of Proceedings of *STP 263*, 1330 (*not to be published*).

Goldthwaite, William H.
See Stang, John H., Goldthwaite, William H., and Dunnington, Bruce W.

Gonser, B. W.
ACR Notes. Published in ASTM BULLETIN, No. 242, Dec., 43.

Goode, Joseph F.
Use of the Immersion-Compression Test in Evaluating and Designing Bituminous Paving Mixtures—Summary of Proceedings of *STP 252*, 1332.

Goodwin, J. G., Rubenstein, L. S., and Shubert, F. L.
The Effects of Heat Treatments on the Tensile and Corrosion Properties of Zircaloy-2—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 272*).

Gordon, B. B.
Extended Repetitive Plate Load Tests for the Evaluation and Design of Concrete Pavements—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 52—Summary of Proceedings of *STP 254*, 1335.

Gouza, J. J.
See Eysenbach, G. E. and Gouza, J. J.

Graham, A. K. and Pinkerton, H. L.
Standardizing the Preparation of Electrodeposits on Test Panels for Corrosion Testing. Report of Committee B-8 (appendix), 317 (*R*).

Graham, R. D. and Miller, D. J.
Marine Exposure Tests of Pressure-Treated Douglas Fir and Southern Pine—Symposium on Treated Wood for Marine Use—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 275*).

Grandolfi, M.
See Sowers, G. F., Vesic, A., and Grandolfi, M.

Grant, N. J.
See Levingston, H. L. and Grant, N. J.

Green, J.
See Peeler, R. L. and Green, J.

Greenfeld, S. H.
See Mertens, E. W. and Greenfeld, S. H.

Grieger, G. R.
Instrumental Modifications for Routine Electron Diffractometry—Summary of Proceedings of *STP 262*, 1072.
Structure Analysis with Routine Electron Diffraction—Summary of Proceedings of *STP 262*, 1072.

Griffin, R. L.
See Simpson, W. C., Miles, T. K., and Griffin, R. L.

Griffiths, K. K.
See Howard, E. L., Griffiths, K. K., and Morton, W. E.

Grover, H. J.
An Observation Concerning the Cycle Ratio in Cumulative Damage—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 274*).
Recent Survey of Current Activities on Fatigue. Published in ASTM BULLETIN, No. 235, Jan., 26. Report of Subcommittee III on Survey of ASTM Committee E-9 on Fatigue.

Grunewald, Max E.
Discussion, 1089.

Guill, J. H. and Moteff, J.
Dosimetry in Europe and the USSR—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 276*).

Gulbransen, Earle A. and Copan, Thomas P.
Electron Microscopy and Electron Difffraction Studies of Oxide Films Formed on Iron in Water and Oxygen Atmospheres—Summary of Proceedings of *STP 256*, 1336.

Gutzeit, Gregoire
Chemical Reactions—Summary of Proceedings of *STP 265*, 1073.
Patent Situation—Summary of Proceedings of *STP 265*, 1073.

H

Haack, L. C.
Manufacture and Application of Asphalt Roofing — Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 280*).

Haefner, K., Lahr, A. F., Meinhart, W. L., and Kanter, J. J.
Property Relationships of Some Cast and Forged Chromium - Manganese - Nickel - Nitrogen Steels Containing 18 per Cent Chromium, 804 (*R*) Disc., 815.

Hagemaier, D. J.
Mobile Field Testing of Missiles and Aircraft —Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 278*).

Hagemaier, D. J.
See Hitt, W. C. and Hagemaier, D. J.

Hahn, K. F.
Lap Shear and Creep Testing of Metal-Adhesive Bonds in Germany—Symposium on Adhesion and Adhesives—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 271*).

Haire, E. T.
Quantitative and Systematic Attack on Fatigue—Symposium on Fatigue in Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 274*).

Hall, K. L., Klaver, R. F., Carroll, J. G., and Bolt, R. O.
Reactor Monitoring by Calorimetry—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be issued in STP 276*).

Halloway, J.
See Criscuolo, E. L., Polansky, D., Halloway, J., and Dyer, C. H.

Hamacker, J. C., Jr.
Discussion, 877.

Hamburger, Walter, J.
The Need to Know. Published in ASTM BULLETIN, No. 238, May 23.

Hamilton, J. J. and Crawford, C. B.
Improved Determination of Preconsolidation Pressure of a Sensitive Clay—Summary of Proceedings of *STP 254*, 1335.

Haney, F. H.
See Butterfield, W. L. and Haney, F. H.

Hanly, F. J.
See Furby, N. W. and Hanly, F. J.

Hanrahan, F. J.
Quality Control and Inspection—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 275*).

Hansen, W. C.
Actions of Calcium Sulfate and Admixtures in Portland Cement—Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 266*).
Discussion, 1119.
Release of Alkalies by Sands and Admixtures in Portland Cement Mortars. Published in ASTM BULLETIN, No. 236, Feb., 35 (TP 45).

Hardrath, H. F. and Naumann, E. C.
Variable Amplitude Fatigue Tests of Aluminum Alloy Specimens—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 274*).

Harris, Jay C.
Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents (Supplement) (*issued as STP 150-C*).
Committee Work and Fundamental Research. Published in ASTM BULLETIN, No. 238, May, 18.

Harris, J. C. and Anderson, R. M.
Removal of Radiotagged Fatty Soil from a Glass Surface—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 268*).

Hartwig, F. J.
Dry Pressing Ceramic Fuel Elements to Close Tolerances—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 276*).

Harwood, Julius J.
The Mechanism of Stress-Corrosion Cracking of Austenitic Stainless Steels. Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels (*published in STP 264*).

Hatch, H. P.
Discussion. Published in ASTM BULLETIN, No. 235, Jan., 41 (TP 11).

Hawkes, Paul E. and Ek, C. Harold
A Machine for Evaluation of High-Temperature Alloys Under Combined Static and Dynamic Stresses. Published in ASTM BULLETIN, No. 235, Jan., 46 (TP 16). Disc., 51 (TP 21).

Hayes, J. G.
Glass Fiber Felt Roofing Products—Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 280*).

Hecker, J. G., Jr.
See Best, George E., Hecker, J. G., Jr., McGrew, J. W., and VandenBerg, R. V.

Hein, T. C. and Schmidt, R. J.
Density Changes in Asphalt Pavement Core Samples—Summary of Proceedings of *STP 252*, 1333.

Heintz, R. M. and Kadoch, K.
The Ram Strati-Charge Engine—Symposium on Air Pollution Control—Summary of Proceedings of Third Pacific Area Meeting, 55 (*publication pending*).

Heller, Robert A.
Discussion, 659, 675.

Hemme, J. M., Jr.,
See Tuthill, L. H., Adams, R. F., and Hemme, J. M.

Hemphill, J. M.
Introduction—Summary of Proceedings of *STP 258*, 1331.

Henrikson, K. G.
See Sharpe, R. Q. and Henrikson, K. G.

Hensley, J. W. and Inks, C. G.
Calcium-45-Tagged Clay as a Detergency Test Soil—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 268*).

Hernandez, H. P.
See Birmingham, B. W., Chelton, D. B., Mann, D. B., and Hernandez, H. P.

Herzog, S. H. and Donaldson, W.
Part I—Ablation and Erosion in the Rocket Environment and Part II—Ablation and Erosion in the Missile Environment—Symposium on Thermal Ablation (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 279*).

Hibler, G. P.
See Levy, D. F., Fassnacht, F. E., Hibler, G. P., Umbach, R. D., and Gagle, D. W.

Hickmott, R. L.
Removal Dose as an Environmental Meassurement of X-rays and Gamma Rays—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 276*).

Higginson, E. C. and Wallace, G. B.
Control Testing for Separation of Lightweight Material from Aggregate. Published in ASTM BULLETIN, No. 243, Jan., 1960, p. 60 (TP 18).

Hill, A. C.
See Pack, M. R., Hill, A. C., and Transtrum, L. G.

Hitt, W. C. and Hagemaier, D. J.
Radiography of Weldments in Motion—Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 278*).

Hochman, Harry
See Drisko, R. W. and Hochman, Harry
See Roe, Thorndyke and Hochman, Harry
See Vind, H. P. and Hochman, Harry

Hockett, J. E.
Compression Testing at Constant True Strain Rates, 1309. Disc., 1317.

Hodgins, P. T.
The Effect of Brick Facial Texture Upon the Initial Rate of Absorption. Published in ASTM BULLETIN, No. 241, Oct., 27 (TP 191).

Hoffman, E. E.
The Solubility of Nitrogen and Oxygen in Lithium and Methods of Lithium Purification—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 272*).

Holden, A. N.
Concluding Remarks—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 276*).

Holden, F. C., Douglass, R. W., and Jaffee, R. I.
Tensile Properties of the Platinum-Group Metals—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 272*).

Holden, F. C., Jaffee, R. I., and Schwartzberg, F. R.
High Temperature Mechanical Properties of Tantalum—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 272*).

Holmberg, M. E.
See Brooks, W. B. and Holmberg, M. E.

Holstein, J. J.
Lateral Shear Tests of a Light Gage Steel Building—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*publication pending*).

Holt, J. M.
Discussion, 733.

Holtz, W. G.
See Ellis, W. and Holtz, W. G.

Honstead, J. F.
See Schwendiman, L. C., Brown, R. E., Honstead, J. F., Linderoth, C. E., and Pearce, D. W.

Housel, W. S.
Dynamic and Static Resistance of Cohesive Soil—1846 to 1958—Summary of Proceedings of *STP 254*, 1335.

Hove, J. E.
Beryllium Oxide for Nuclear Applications—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 276*).

Howard, E. L., Griffiths, K. K., and Morton, W. E.
Field Experience Using Water Reducers in Ready Mixed Concrete—Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 266*).

Howes, J. E., Jr., Brown, C. T., Sunderman, D. N., Pobereskin, M., and Schall, P.
A Radiometric Method for the Determination of Magnesium Oxide in Portland Cement—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 268*).

Hrenoff, M. K.
See Strait, L. A. and Hrenoff, M. K.

Hribar V. F. and Dowell, A. M.
Evaluation of Reinforced Plastics for High Temperature Structural Applications—Symposium on Reinforced Plastics—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 279*).

Hsiao, C. M.
See Kasak, A., Hsiao, C. M., and Dulis, E. J.

Hubis, W.
See Coggeshall, N. D., and Hubis, W.

Hull, F. C.
Discussion, 815.

Hurd, J. N.
See Beaver, W. W., O'Rourke, R. G., and Hurd, J. N.

Hurd, J. N., O'Rourke, R. G., and Beaver, W. W.
The Stress-Rupture and Creep Properties of QMV Beryllium Metal—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 272*).

Hurwitz, J. K.
The Matrix Effect in Excitation—Summary of Proceedings of *STP 259*, 1338.

Hveem, F. N.
Comments on ASTM Standards for Highway Materials—Symposium on Standards—Summary of Proceedings of Third Pacific Area Meeting, 55 (*publication pending*).

Hveem, F. N., Zube, Ernst, and Skog, J. B.
Progress Report on the Zaca-Wigmore Experimental Asphalt Test Project—Symposium on Road and Paving Materials (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 277*).

Hymes, L. C.
See Noland, R. A., Walker, D. E., and Hymes, L. C.

I

Illg, Walter
Discussion, 752.
See McEvily, Arthur, J., Jr. and Illg, Walter

Ingberg, S. H.
Compression Tests of Wood at Elevated Temperatures, 1320.

Ingle, G. W.
Summation—Summary of Proceedings of *STP 258*, 1331.

Inks, C. G.
See Hensley, J. W. and Inks, C. G.

Irick, P. E.
Fundamental Statistical Concepts in Testing—Summary of Proceedings of *STP 252*, 1333.

Irwin, G. R.
Discussion, 914.

J

Jaffee, R. I.
Discussion, 1013, 1071.
See Holden, F. C., Douglass, R. W., and Jaffee, R. I.
See Holden, F. C., Jaffee, R. I., and Schwartzberg, F. R.
See Simmons, W. F., Sirois, B. J., Williams, D. N., and Jaffee, R. I.

Janis, E. C.
Elevated Temperature Resistant Metal-to-Metal Adhesives Derived from Organic-Inorganic Polymer Systems—Symposium on Adhesion and Adhesives—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 271*).

John, R. R. and Bade, W. M.
Testing of Reinforced Plastics Under Simulated Re-Entry Conditions, Part I—Plasma Jet Calibration—Symposium on Thermal Ablation (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 279*).

John, R. R. and Schick, H. L.
Testing of Reinforced Plastics Under Simulated Re-Entry Conditions, Part II—Experimental Determination of the Steady-State Heat of Ablation—Symposium on Thermal Ablation (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 279*).

Johnson, A. I.
See Prill, R. C. and Johnson, A. I.

Johnson, A. I. and Morris, D. A.
Correlation of Atterberg Limits with Geology for Deep Cores from Subsidence Areas in California—Summary of Proceedings of *STP 254*, 1335.

Johnson, C. A.
Electrical Insulating Liquids 1916–1959—The Story of Subcommittee IV, Committee D-9. Published in ASTM Bulletin, No. 238, May, 25.

Johnson, J. W.
Screw-Holding Ability of Western Woods: Effects of Test Variables—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Meeting, 46 (*to be published in STP 275*).

Johnson, K. R.
See Wholers, H. C., Kass, T. E., and Johnson, K. R.

Johnson, P. V. and Soderstrum, W. K.
Relation of Current ASTM Specifications to Properties of Lightweight Clay Masonry Units—Symposium on Masonry Materials—Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*).

Jones, D. R.
See Jursch, C. F., Pinotti, P. L., and Jones, D. R.

Jones, M. H.
See Espey, G. B., Jones, M. H., and Brown, Jr., W. F.

Joslin, J. G. and Davis, H. D.
Ohio Adopts the One-Point Mechanical Method for Determining the Liquid Limit of Soils—Summary of Proceedings of *STP 254*, 1335.

Jursch, C. F., Pinotti, P. L., and Jones D. R.
Radioactive Ring Wear Testing in Railroad Diesel Locomotives—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 268*).

Juve, A. E.
Mooney Cure Tests for Calculating Curing Times. Published in ASTM Bulletin, No. 239, July, 71 (TP 151).

K

Kadoch, K.
See Heintz, R. M. and Kadoch, K.

Kall, H. L.
Test Methods Used for Zirconium and Zirconium Alloy in the Naval Reactor Program—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 272*).

Kammerer, C. C.

Ultrasonic Inspection of Adhesion Bonded Aluminum Sandwich Structures—Symposium on Nondestructive Testing in the Missile Industry—Summary of Third Pacific Area Meeting, 41 (*to be published in STP 278*).

Kampf, Leo

Discussion. Published in ASTM BULLETIN, No. 240, Sept., 45 (TP 175).

Kane, J. R.

Use of Color Photography for Product Specification and Control—Summary of Proceedings of *STP 258*, 1331 (*not to be published*).

Kanter, J. J.

See Haefner, K., Lahr, A. F., Meinhart, W. L., and Kanter, J. J.

Kantro, D. L.

See Brunauer, Stephen, Copeland, L. E., Kantro, D. L., Weise, C. H., and Schultz, Edith G.

Karol, R. H.

Automation in Consolidation Testing—Summary of Proceedings of *STP 254*, 1335.

Kasak, A., Hsiao, C. M., and Dulis, E. J.

Relationships Between Composition and Properties of Austenitic Chromium-Manganese-Carbon-Nitrogen Stainless Steels, 786. Disc., 802.

Kass, T. E.

See Wohlers, H. C., Kass, T. E., and Johnson, K. R.

Katz, W. E.

Operation of Batch-Type and Continuous Electric Membrane Demineralizers on the Municipal Water Supply of Coalinga, Calif.—Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 273*).

Kaups, Taavi

Discussion. Published in ASTM BULLETIN, No. 239, July, 79 (TP 159).

Kaye, W. I.

Spectroscopy in the Region 175–200 mμ—Symposium on Spectroscopy (Session III)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 269*).

Keeney, W. D.

Wood-Pole Type Buildings—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 275*).

Kelch, N. W.

Field Test Specimens of Mortar and Grout for Reinforced Grouted Brick Masonry—Symposium on Masonry Materials—Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*).

Testing Reinforced Grouted Brick Masonry and Also Hollow Concrete Masonry Wall Panels to Determine Values in Diagonal Tension and Shear—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*publication pending*).

Keller, G. R.

Is Hydraulics Out of This World?—Symposium on Hydraulic Fluids (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 267*).

Kelley, R. M., Blank, E. W., Thompson, W. E., and Fine, R.

The Determination of Alkyl Aryl Sulfonates by Ultraviolet Absorption. Published in ASTM BULLETIN, No. 237, April, 70 (TP 90) (*R*).

Kemp, J. W.

The Future of X-ray Fluorescence Instrumentation—Symposium on Spectroscopy (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 269*).

See Andermann, G. and Kemp, J. W.

Kennedy, Ralph G.

Discussion, 658, 1278.

Kennedy, T. B.

Discussion, 1108.

Kerlin, E. E.

See Newell, D. M., Kerlin, E. E., Bauerlein, R. R., and Barrows, R. F.

Kesler, Clyde E.

Effect of Length to Diameter Ratio on Compressive Strength—An ASTM Cooperative Investigation, 1216. Disc., 1229.

Kester, B. E.

Acetate Processed Portland Cement, 1111. Disc., 1118.

Kidder, A. H.

Discussion, 1260.

King, H. A.

Testing of Materials Under Various Ablative Conditions—Symposium on Thermal Ablation (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 279*).

King, H. F. and Coil, J. A.

Hydraulic Fluids for Use in High Pressure Shipboard Equipment—Symposium on Hydraulic Fluids (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 267*).

Kinney, J. E.
Cities Can and Must Provide Municipal Waste Treatment, Not Sewage Treatment—Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 273*).

Kinsey, H. V.
The Creep and Rupture Properties of Three Cast Copper-Base Alloys Between 550 and 650 F (290 and 345 C), 940.

Klaver, R. F.
See Hall, K. L., Klaver, R. F., Carroll, J. G., and Bolt, R. O.

Kleiman, W. F.
See Weber, W. G., Jr. and Kleiman, W. F.

Klein, Alexander
See Polivka, Milos and Klein, Alexander

Klieger, Paul
Discussion. Published in ASTM Bulletin, No. 235, Jan., 41 (TP 11).

Klier, E. P.
Discussion, 875.

Klier, E. P. and Gazzara, C. P.
Stress-Strain Properties of Selected Titanium Metals, 1015 (*R*). Disc., 1029.

Koenig, J. H.
Electronic Ceramics (Committee C-21 on Ceramic Whitewares and Related Products)—Symposium on Materials in the Electronics Industry—Summary of Proceedings of Third Pacific Area Meeting, 43 (*not to be published*).

Kolloff, R. H.
Analysis of Commercial Sodium Tripolyphosphate by Reverse Flow Ion Exchange Chromatography. Published in ASTM Bulletin, No. 237, April, 74 (TP 94) (*R*).

Kovacich, S. A.
See Peeler, R. L. and Kovacich, S. A.

Kramer, K. H. and Steiner, J. E.
Effect of Composition and Processing Variables on Some Mechanical Properties of Large Nickel-Molybdenum-Vanadium Rotor Forgings, 916.

Krieg, Abraham
Processing Procedures—Summary of Proceedings of *STP 265*, 1073.

Kropschot, R. H.
See McClintock, R. M., Van Gundy, D. A., and Kropschot, R. H.

Kuenning, William H.
Discussion. Published in ASTM Bulletin, No. 241, Oct., 26 (TP 190).

Kula, Erick, B., and Larson, Frank R.
Effect of Specimen Taper on Determination of Elongation in the Tension Test. Published in ASTM Bulletin, No. 238, May, 58 (TP 126). Disc., No. 241, Oct., 37 (TP 201).

Kuranz, John
Measurement of Moisture and Density in Soils by the Nuclear Method—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 268*).

L

Lahr, A. F.
See Haefner, K., Lahr, A. F., Meinhart, W. L., and Kanter, J. J.

Lappala, R. P., Yaeger, L. L., and Bjorksten, Johan
Present Status of Solar Distillation of Saline Water with Particular Regard to Material Problems—Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 273*).

LaPorta, J. A.
See Parker, E. G., Winter, R. M., and LaPorta, J. A.

LaQue, F. L.
ASTM, Materials, and Progress. Published in ASTM Bulletin, No. 242, Dec., 20.
ASTM Views on Engineering Education—Summary of Proceedings of *STP 263*, 1330.
Introductory Summary. Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels (*published in STP 264*).

Larrabee, C. P.
See Copson, H. R. and Larrabee, C. P.

Larson, Frank R.
Discussion, 1307.
See Kula, Eric B. and Larson, Frank R.

Lashof, T. W.
See Mandel, John and Lashof, T. W.

Laskie, J. R.
The Use of Molded Activated Carbon Getters in Sealed Electrical Instruments. Published in ASTM Bulletin, No. 241, Oct., 41 (TP 205). (Technical Note.)

Lazan, Benjamin J.
Discussion, 685, 709, 733.

Lee, Henry
Compatibility of Magnet Wire Insulations and Epoxy Encapsulating Resins—Symposium on Electrical Insulating Materials—Summary of Proceedings of Third Pacific Area Meeting, 41 (*publication pending*).

Legget, R. F.
Introduction—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 275*).
Common Sense and Building Materials. Published in ASTM Bulletin, No. 237, April, 36.

Lehmann, H. L. and Adam, Verdi
Application of the Marshall Method to Hot-Mix Design—Summary of Proceedings of *STP 252*, 1332.

Lent, W. E.
See Gates, L. E. and Lent, W. E.

Lenzen, T. L.
Foreign Petroleum—Petroleum and Chemical Industry Luncheon Address—Summary of Proceedings of Third Pacific Area Meeting, 37 (*not to be published*).

Leonards, G. A. and Ramiah, B. K.
Time Effects on the Consolidation of Clays—Summary of Proceedings of *STP 254*, 1335.

Lepper, Henry A., Jr.
Viewpoint of the Civil Engineer—Summary of Proceedings of *STP 263*, 1330.

Levingston, H. L. and Grant, N. J.
Deoxidation and Denitrification Studies of Chromium and the Effect on Low Temperature Properties—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 272*).

Levy, D. F., Fassnacht, F. E., Hibler, G. P., Umbach, R. D., and Gagle, D. W.
Fundamental Viscosity *versus* Saybolt Furol Viscosity for Refinery Control of Cutback Asphalt—Summary of Proceedings of *STP 252*, 1333.

Lewis, J. E.
Experiences with Activation in the Analysis of Aluminum—Summary of Proceedings of *STP 261*, 1339.

Linderoth, C. E.
See Schwendiman, L. C., Brown, R. E., Honstead, J. F., Linderoth, C. E., and Pearce, D. W.

Lipsitt, H. A.
See Baird, R. B., Forbes, F. W., and Lipsitt, H. A.

Lipsitt, H. A., Forbes, F. W., and Baird, R. B.
Crack Propagation in Cold-Rolled Aluminum Sheet, 734 (*R*). Disc., 748.

Lischer, A. A.
Development Fatigue Testing of a Helicopter Rotor Blade—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 274*).

Lominska, C. A.
Exposure Fence Testing of Metal Protective Paints—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).

Long, Augustus C.
Business Man Abroad. Published in ASTM Bulletin, No. 237, April, 28.

Long, R. W.
See Meese, R. H. and Long, R. W.

Longworth, Jack and Stieda, C. K. A.
Strength Studies of Scarf Joints in Laminated Wood Beams—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 275*).

Loy, W. E., Jr.
The Effects of Gamma Radiation on Some Electrical Properties of Teflon—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 276*).

Lubahn, J. D.
Experimental Determination of Energy Release Rate for Notch Bending and Notch Tension, 885 (*R*). Disc., 914.
The Effect of Temperature Interruption on Anelastic Creep. Published in ASTM Bulletin, No. 239, July, 61 (TP 141). (Technical Note.) Disc., No. 243, Jan., 1960, 54 (TP 12).

Lula, R. A.
Discussion, 659, 785.

Lundeen, R. W.
See Moore, R. J., Boscia, T. D., and Lundeen, R. W.

Lysaght, Vincent E.
Discussion, 1278.

M

Maag, Ernst
The Renaissance of Wood in School Building Construction in California—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 275*).

MacGregor, C. W.
Discussion, 1317.

Mackey, Royal V., Jr.
See Cahn, Harold L. and Mackey, Royal V., Jr.

Mackintosh, Albyn
Materials for Reinforced Masonry Construction—Symposium on Masonry Materials—Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*).

MacPherson, D. R. and Fischer, H. C.
The Effect of Water-Reducing Admixtures and Set-Retarding Admixtures on the Properties of Hardened Concrete—Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 266*).

Maddin, C. M. and Rosene, R. B.
Identification by Instrumental Methods of Chemical Compounds in Water-Formed Deposits—Summary of Proceedings of *STP 256*, 1336.

Main, R. M.
Tracer Methods for Rapid Determination of the Uniformity of Mixing—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 268*).

Mancini, M. A.
See Degering, E. F., Caldarella, G. J., and Mancini, M. A.

Mandel, J.
Discussion, 1277.
The Theory of Extreme Values. Published in ASTM Bulletin, No. 236, Feb., 29 (TP 39) (*R*).

Mandel, John and Lashof, T. W.
The Interlaboratory Evaluation of Testing Methods. Published in ASTM Bulletin, No. 239, July, 53 (TP 133).

Manjoine, M. J., France, L. L., and Begley, R. T.
Universal High Temperature Testing Machine for Vacuum or Controlled Atmosphere—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 272*).

Mann, D. B.
See Birmingham, B. W., Chelton, D. B., Mann, D. B., and Hernandez, H. P.

Manov, G. G. and Beaufait, L. J.
Instrumental Methods of Analysis—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 268*).

Marble, J. D.
Discussion, 658.

Mardulier, F. J.
Discussion, 1087, 1118.

Margoshes, M.
Some Properties of New or Modified Excitation Sources—Summary of Proceedings of *STP 259*, 1338.

Marin, J., Borachia, P., and Rimrott, U. A.
The Effect of Stress Cycling on the Static Mechanical Properties of SAE 4340 Steel, 662 (*R*). Disc., 673.

Marin, Joseph
Some Proposed Changes in Standards Based Upon Research in Materials—Symposium on Standards—Summary of Proceedings of Third Pacific Area Meeting, 55 (*publication pending*).

Mark, Herman F.
New Polymers—New Problems. Published in ASTM Bulletin, No. 241, Oct., 6 (synopsis); No. 245, April, 1960. (Marburg Lecture.) (*published as separate pamphlet*)

Marshall, A. C.
Effect of Normal Aging on Physical Properties of Glass Fabric-Plastic Honeycomb Core—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 270*).

Martin, D. E. and Brinn, J.
Some Observations on the Plastic Work Required to Fracture Stainless Steel Under Cyclic Loading, 677 (*R*). Disc., 685.

Mather, Bryant
Discussion, 1202, 1214.
See Pepper, Leonard and Mather, Bryant

May, T. K.
Determining the Useful Strength of Yard Lumber of the Common Grade—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 275*).

McCarthy, W. R.
Test Procedure for Obtaining Mechanical Properties of Filament Wound Materials—Symposium on Reinforced Plastics—Summary of Proccedings of Third Pacific Area Meeting, 47 (*to be published in STP 279*).

McCartney, R. F. and Trozzo, P. S.
Electron Metallography of Neutron-Irradiated Steels—Summary of Proceedings of *STP 262*, 1072.

McClintock, R. M., Van Gundy, D. A., and Kropschot, R. H.
Low-Temperature Tensile Properties of Copper and Four Bronzes. Published in ASTM Bulletin, No. 240, Sept., 47 (TP 177).

McCormick, F. J.
See Raville, M. E. and McCormick, F. J.

McCoy, Ernest E., Jr.
Discussion. Published in ASTM Bulletin, No. 236, Feb., 47 (TP 57).

McCoy, Paul
Sound Standards Stimulate Manufacturing—Symposium on Standards—Summary of Proceedings of Third Pacific Area Meeting, 55 (*publication pending*)

McCoy, W. J.
Discussion, 1119.

McCrum, N. G.
Torsion Pendulum Method for Determining Crystallinity and Void Content of Tetrafluoroethylene Resins. Published in ASTM Bulletin, No. 242, Dec., 80 (TP 240).

McCulloch, Alex
Development of Loading Spectra for Testing Aircraft Components and Structures—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 274*).

McCullough, H. M.
Columbium and Tantalum Fabrication—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 272*).

McDowell, D. W. Jr.
Discussion, 833.

McEvily, A. J., Jr. and Illg, Walter
A Method for Predicting the Rate of Fatigue Crack Propagation—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 274*).
Discussion, 752.

McGarry, F. J.
Resin-Glass Bond Characteristics. Published in ASTM Bulletin, No. 235, Jan., 63 (TP 33).
See Chambers, R. E. and McGarry, F. J.
See Desai, M. B. and McGarry, F. J.

McGrew, J. W.
See Best, George E., Hecker, J. G., Jr., McGrew, J. W., and VandenBerg, R. V.

McLeod, Norman W.
Practical Significance of Tests on Asphalt Cement—Summary of Proceedings of *STP 252*, 1333.
Void Requirements for Dense-Graded Bituminous Paving Mixtures—Summary of Proceedings of *STP 252*, 1332.

McMinn, Jack
Soils Classification Triangle Based on the Unified Soil Classification System—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 54—Summary of Proceedings of *STP 254*, 1335.

McMullin, J. G. and Yukawa, S.
See Yukawa, S. and McMullin, J. G.

McMurtry, C. H.
See Taylor, K. M. and McMurtry, C. H.

McNally, J. R., Jr.
Challenges in Atomic Spectroscopy—Symposium on Spectroscopy (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 269*).

McPherson, Archibald T.
Science Fairs, ACR notes. Published in ASTM Bulletin, No. 237, April, 29.

McRae, John L. and Foster, Charles R.
Theory and Application of a Gyratory Testing Machine for Hot-Mix Bituminous Pavement—Summary of Proceedings of *STP 252*, 1332.

Meese, R. H. and Long, R. W.
Triaxial Compression Tests on Soils Using Variable Lateral Pressure—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 54—Summary of Proceedings of *STP 254*, 1335.

Meggers, W. F.
See Scribner, B. F. and Meggers, W. F.

Meinhart, W. L.
See Haefner, K., Lahr, A. F., Meinhart, W. L., and Kanter, J. J.

Meinke, W. W.
Nucleonics in Analysis—Summary of Proceedings of *STP 261*, 1339.

Mellon, M. G. and Bly, D. D.
Bibliography of Spectrophotometric Methods of Analysis for Organic Ions (1959 Supplement) (*issued as STP 125-A*).

Mertens, E. W. and Greenfeld, S. H.
Some Qualitative Effects of Composition and Processing on the Weatherability of Coating Grade Asphalts—Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 280*).

Metzger, Paul D.
A Method of Determining the Demulsibility of Lubricating Oils. Published in ASTM Bulletin, No. 242, Dec., 77 (TP 237).

Metzger, W. H., Jr.
Characteristics of Deposits—Summary of Proceedings of *STP 265*, 1073.

Mielenz, R. C.
Water-Reducing Admixtures and Set-Retarding Admixtures for Concrete: Uses; Specifications; Research Objectives—Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 266*).

Mihalisin, J. R.
Morphology of Phases in High-Temperature Alloys as Revealed by the Electron Microscope—Summary of Proceedings of *STP 262*, 1072.

Mikus, E. B. and Siebert, C. A.
The Effect of Temper-Embrittlement on the Fatigue Properties and Damage Due to Fatigue Overstressing of a 3140 Steel (*R*). Disc., (*publication pending*).

Miles, T. K.
See Simpson, W. C., Miles, T. K., and Griffin, R. L.

Miller, D. J.
See Graham, R. D. and Miller, D. J.

Miller, N. R.
Discussion. Published in ASTM BULLETIN, No. 241, Oct., 40 (TP 204).

Millett, W. H.
Objectives and Activities of Technical Committee N (of ASTM Committee D-2)—Symposium on Hydraulic Fluids (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 267*).

Mills, Blake D., Jr.
Discussion. Published in ASTM BULLETIN, No. 236, Feb., 47 (TP 57).

Minnick, L. John
Fundamental Characteristics of Pulverized Coal Fly Ashes, 1155.

Minor, Carl E.
Correlation of Hveem Stabilometer and Cohesiometer Test Results and Kneading Compactor Densities with Service Records of Bituminous Pavements—Summary of Proceedings of *STP 252*, 1332.
Degradation of Mineral Aggregates—Symposium on Road and Paving Materials (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 277*).

Mitchell, J. E.
Liquid Limit Results from Various Types of Grooving Tools—Summary of Proceedings of *STP 254*, 1335.

Monego, C. J.
See Wing, Paul and Monego, C. J.

Monismith, C. L.
Effect of Temperature on Flexibility Characteristics of Asphaltic Paving Mixtures—Symposium on Road and Paving Materials (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 277*).

Moore, Anna T.
See Rollins, Mary L., de Gruy, Ines V., Tripp, Verne W., and Moore, Anna T.

Moore, R. J., Boscia, T. D., and Lundeen, R. W.
Wind and Weather Summaries for Chemical Plant Design and Air Pollution Control—Symposium on Air Pollution Control—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 281*).

Moorehead, D. R. and Taylor, W. H.
The Sucrose Extraction Method of Determining a Variable Calcium Oxide in Hydrated Lime. Published in ASTM BULLETIN, No. 236, Feb., 45 (TP 55).

Moran, J. J.
See Ruh, E. L., Moran, J. J., and Thompson, R. D.

Morehead, F. F.
Introduction—Summary of Proceedings of *STP 257*, 1074.

Morris, D. A.
See Johnson, A. I. and Morris, D. A.

Morris, H. C.
The Effect of Particle Shape and Texture on the Strength of Noncohesive Aggregates—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 54—Summary of Proceedings of *STP 254*, 1335.
See Ekse, Martin and Morris, H. C.

Morris, J. M., Jr.
Disposal of Oil Field Brines in the Central Valley of California—Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 273*).

Morris, M. D., Ulp, R. B., and Spinna, R. A.
Proposed Universal Standards for the Liquid Limit Tests—Summary of Proceedings of *STP 254*, 1335.

Morrison, G. H.
Metals Analysis by Radioactivation—Summary of Proceedings of *STP 261*, 1339.

Morrow, JoDean
Discussion, 686.

Morton, W. E.
See Howard, E. L., Griffiths, K. K., and Morton, W. E.

Moteff, J.
See Guill, J. H. and Moteff, J.

Mowbray, A. Q.
A Pig Is a Pig Is a Pig. Published in ASTM BULLETIN, No. 241, Oct., 18.
Discussion, 709.

Muccigrosso, A. T.
See Ploetz, G. L. and Muccigrosso, A. T.

Muchnick, Samuel
Surface Chemistry of Adhesion—Symposium on Adhesion and Adhesives—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 271*).

Murphy, Glenn
Nature and Properties of Metals—a Discussion of the Report of ASEE Followup Subcommittee on Materials—Summary of Proceedings of *STP 263*, 1330.

Murphy, J. F.
Suggested Method for Spectrochemical Analysis of Magnesium-Base Alloys by the Point-to-Plane Spark Technique Using a Grating Spectrograph. Report of Committee E-2 (appendix II), 577.

N

Nason, Howard K.
Management's Changing Responsibilities. Published in ASTM BULLETIN, No. 242, Dec., 28.

Naumann, E. C.
See Hardrath, H. F., and Naumann, E. C.

Neisel, R. H.
Prediction of Temperature Rise in Fire Testing of Walls and Partitions. Published in ASTM BULLETIN, No. 239, July, 66 (TP 146).

Nelson, G. A.
Residual Stresses in Heat-Exchanger Tubes. Published in ASTM BULLETIN, No. 240, Sept., 39 (TP 169). (Technical Note.)

Neppiras, E. A.
Techniques and Equipment for Fatigue Testing at Very High Frequencies, 691 (*R*). Disc., 709.

Neville, A. M.
The Influence of the Direction of Loading on the Strength of Concrete Test Cubes. Published in ASTM BULLETIN, No. 239, July, 63 (TP 143).

Newell, D. M., Kerlin, E. E., Bauerlein, R. R., and Barrows, R. F.
Dynamic Radiation Effects Testing Methods—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 276*).

Newman, Sanford B.
Microscopical Studies of Failure of Polymers—Summary of Proceedings of *STP 257*, 1074.

Newton, Clarence J.
False Negative Permanent Strains Observed with Resistance Wire Strain Gages. Published in ASTM BULLETIN, No. 235, Jan., 42 (TP 12). Disc., No. 239, July, 79 (TP 159)

Nicodemus, P. O.
The Significance of Iron in Asbestos Materials Used for Electrical Insulating Purposes. Published in ASTM BULLETIN, No. 237, April, 62 (TP 82). Disc., 67 (TP 87).

Nielsen, N. A.
Introduction—Summary of Proceedings of *STP 262*, 1072.

Nimeroff, I.
Status of ASTM Methods and Standards for Appearance Evaluation—Summary of Proceedings of *STP 258*, 1331.

Noguera, G.
See Davidson, D. T., Noguera, G., and Sheeler, J. B.

Noland, R. A., Walker, D. E., and Hymes, L. C.
The Fabrication of U_3O_8-Al Dispersion Fuel Elements by Extrusion—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 276*).

Novak, E. S.
See Wilson, A. G., Solvason, K. R., and Novak, E. S.

O

Oatman, H. T.
See Caugherty, Betty, Oatman, H. T., and Reen, O. W.

Offutt, J. S.
Discussion, 1108.

O'Kelly, B. M.
Physical Changes in Setting Gypsum Plaster. Published in ASTM BULLETIN, No. 237, April, 55 (TP 75) (*R*). Disc., No. 243, Jan., 1960, 51 (TP 9).

Oliensis, G. L.
Discussion. Published in ASTM BULLETIN, No. 235, Jan., 36 (TP 6).

Olszewski, W. V.
See Doyle, C. D. and Olszewski, W. V.

Ore, E. L.
See Wallace, G. B. and Ore, E. L.

O'Rourke, R. G.
See Beaver, W. W., O'Rourke, R. G., and Hurd, J. N.
See Hurd, J. N., O'Rourke, R. G., and Beaver, W. W.

P

Pabst, William R., Jr.
Standardization in India. Published in ASTM BULLETIN, No. 242, Dec., 31.

Pack, M. R., Hill, A. C., and Transtrum, L. G.

Determination of Gaseous and Particulate Inorganic fluorides in the Atmosphere—Symposium on Air Pollution Control—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 281*).

Padlog, Joseph and Rattinger, Ivan

Low-Cycle Fatigue Strength of Pressurized Components—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 274*).

Panian, F. C. and Van Valkenburg, H. E.

Development of ASTM Standard Reference Blocks for Ultrasonic Inspection, 1237 (*R*). Disc., 1259.

Papirno, Ralph

See Tramposch, Herbert, Papirno, Ralph, and Gerard, George

Parker, F. G., Winter, R. M., and LaPorta, J. A.

Magnetic Properties of Copper Alloys. Report of Committee B-5 (appendix III), 230 (*R*).

Parkinson, W. W. and Binder, D.

Post-Irradiation Oxidation and Molecular Weight Changes in Polysterene and Poly-(methyl methacrylate)—Symposium on Post-Irradiation Effects in Polymers—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 276*).

Pearce, D. W.

See Schwendiman, L. C., Brown, R. E., Honstead, J. F., Linderoth, C. E., and Pearce, D. W.

Peckner, D.

Segregation in Aluminum—13 Per Cent Uranium Castings. Published in ASTM Bulletin, No. 242, Dec., 51 (TP 211).

Peeler, R. L. and Green, J.

Measurement of Bulk Modulus of Hydraulic Fluids. Published in ASTM Bulletin, No. 235, Jan., 51 (TP 21) (*R*).

Peeler, R. L. and Kovacich, S. A.

Development and Testing of Aircraft Hydraulic Fluids—Symposium on Hydraulic Fluids (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 267*).

Pellier, L.

A Simple Application of the Carbon Replica Technique for Electron Metallography—Summary of Proceedings of *STP 262*, 1072.

Pellissier, G. E.

Simplified Electron Metallography of Steels—Summary of Proceedings of *STP 262*, 1072.

Pepper, Leonard and Mather, Bryant

Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction, 1178 (*R*). Disc., 1202.

Perry, H. A.

Ablative Environments—Introductory Remarks—Symposium on Thermal Ablation (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 279*).

Introductory Remarks—Problems in Testing and Specifying Reinforced Plastics—Symposium on Reinforced Plastics—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 279*).

See Silver, I., Erickson, P. W., and Perry, H. A.

Peterson, R. E.

Discussion, 750.

Phelan, Paul F.

Discussion. Published in ASTM Bulletin, No. 235, Jan., 36 (TP 6).

Pinkerton, H. L.

See Graham, A. K. and Pinkerton, H. L.

Pinotti, P. L.

See Jursch, C. F., Pinotti, P. L., and Jones, D. R.

Pittman, C. U. and Donegan, J. W.

Industrial Pitches—Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 54 (*publication pending*).

Ploetz, G. L. and Muccigrosso, A. T.

Forming of Ceramic Particles for Nuclear Fuels, Control and Burnable Poison Dispersion Components—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 276*).

Pobereskin, M.

Permeability Tests for Organic Sheet Materials—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 268*).

See Howes, J. E., Jr., Brown, C. T., Sunderman, D. N., Pobereskin, M., and Schall, P.

Polansky, D.

See Criscuolo, E. L., Polansky, D., Halloway, J., and Dyer, C. H.

Polivka, Milos and Klein, Alexander

Effect of Water-Reducing Admixtures and Set-Retarding Admixtures as Influenced by Portland Cement Composition—Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 266*).

Potts, W. J.
Some of the Problems Facing Chemical Infrared Spectroscopy—Symposium on Spectroscopy (Session V)—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 269*).

Powers, T. C.
Discussion, 1202.

Preuss, L. E.
Tracer Techniques for Very Thin Vacuum Evaporated Metal Films—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 268*).

Prill, R. C. and Johnson, A. I.
The Effect of Temperature on Moisture Contents as Determined by Centrifuge and Tension Techniques—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 54—Summary of Proceedings of *STP 254*, 1335.

Prior, M. R. and Adams, A. B.
Introduction to Producers' Papers on Water-Reducing Admixtures and Set-Retarding Admixtures for Concrete—Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 266*).

Pulsifer, Verne
The Annealing Behavior of 70-30 Brass Strip (Cartridge Brass, 70 per Cent), 930 (*R*).

R

Raber, Charles T.
Discussion, 1108.

Rader, L. F.
General Factors in Design of Bituminous Paving Mixtures—Summary of Proceedings of *STP 252*, 1332.

Ramiah, B. K.
See Leonards, G. A. and Ramiah, B. K.

Ratliff, George D., Jr.
See Benjamin, Irwin A. and Ratliff, George D., Jr.

Rattinger, Ivan
See Padlog, Joseph and Rattinger, Ivan

Raville, M. E. and McCormick, F. J.
Stresses in Deep Beams Subjected to Central and Third-Point Loading, 1230.

Rea, D. G.
Molecular Spectroscopy in the USSR—Symposium on Spectroscopy (Session VI)—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 269*).

Reen, O. W.
See Caugherty, Betty, Oatman, H. T., and Reen, O. W.

Reese, D. J.
See Trojan, Paul K., Flinn, R. A., and Reese, D. J.

Reese, J. P.
Service Experience of Sandwich Construction—Some Approaches to Problems in Aircraft Applications—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 270*).

Repko, A. J.
See Epsey, G. B., Repko, A. J., and Brown, W. F., Jr.

Rice, J. K.
Deposit Identification—First Step Toward Understanding a Water Problem—Summary of Proceedings of *STP 256*, 1336.

Richards, Oscar W.
Measurement with Phase and Interference Microscopes—Summary of Proceedings of *STP 257*, 1074.

Richman, R. B.
Improvements in Water Treatment for Once-Through Reactor Cooling—Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 273*).

Richmond, Owen
Discussion, 676.

Rider, D. K.
Temperature Rise in Structures Due to Solar Heating—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 270*).

Riley, W. C.
Graphite as a Fuel Matrix—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 276*).

Rimrott, U. A.
See Marin, J., Borachia, P., and Rimrott, U. A.

Risk, T. H.
Automotive Central Hydraulic Systems and Their Fluid Requirements—Symposium on Hydraulic Fluids (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 267*).

Rittenhouse, John
Discussion, 983.

Robey, R. F.
Ten Years of Aid to Applied Absorption Spectroscopy by Committee E-13—Symposium on Spectroscopy (Session V)—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 269*).

Robinson, Elmer
The Air Pollution Potential of California Coastal Climate—Symposium on Air Pollution Control—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 281*).

Roe, Thorndyke and Hochman, Harry
Use of Small Panels in the Screening of Potential Wood Preservatives—Symposium on Treated Wood for Marine Use—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 275*).

Rogers, P. L.
Testing Masonry Mortar for Effluorescence—Symposium on Masonry Materials—Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*).
A Method of Test for Potential Effluorescence of Masonry Mortar. Published in ASTM BULLETIN, No. 235, Jan., 31 (TP 1). Disc., No. 240, Sept., 45 (TP 175).

Rollins, Mary L., deGruy, Ines V., Tripp, Verne W., and Moore, Anna T.
Microscopical Evaluation of Cotton Finishing Treatments—Summary of Proceedings of *STP 257*, 1074.

Romualdi, J. P. and D'Appolonia, E.
Effect of Internal Heating on the Fatigue Life of Titanium, 723. Disc., 731.

Rosene, R. B.
See Maddin, C. M. and Rosene, R. B.

Rosenblum, C.
Refereeing by Isotope Dilution Methods—Summary of Proceedings of *STP 261*, 1339.

Rostler, F. S. and White, R. M.
Influence of Chemical Composition of Asphalts on Performance, Particularly Durability—Symposium on Road and Paving Materials (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 277*).

Rowand, H. H.
Spray Flammability Test for Hydraulic Fluids—Symposium on Hydraulic Fluids (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 267*).

Royen, Markus
A Study of the Accelerated Aging of Vinyl Plastic Compounds in a Modified Testing Oven. Published in ASTM BULLETIN, No. 243, Jan., 1960, 43 (TP 1).

Rubenstein, L. S.
See Goodwin, J. G., Rubenstein, L. S., and Shubert, F. L.

Rudin, A.
See Birks, A. M. and Rudin, A.

Ruh, E. L., Moran, J. J., and Thompson, R. D.
Measurement Problems in the Instrument and Laboratory Apparatus Field. Published in ASTM BULLETIN, No. 240, Sept., 31 (TP 161).

Ruprecht, W. J.
Discussion, 879.

S

Sachs, G., Sell, R., and Brown, Jr., W. F.
Tension, Compression, and Fatigue Properties of Several Steels for Aircraft Bearing Applications, 635 (*R*). Disc., 658.

Safranek, W. H.
Applications—Summary of Proceedings of *STP 265*, 1073.

Samans, C. H.
See Tisinai, G. F. and Samans, C. H.

Sands, R. H.
The Unpaired Electron via EPR—Symposium on Spectroscopy (Session IV)—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 269*).

Saubestre, E. B.
Advantages and Limitations—Summary of Proceedings of *STP 265*, 1073.

Schall, P.
See Howes, J. E., Jr., Brown, C. T., Sunderman, D. N., Pobereskin, M., and Schall, P.

Schaub, J. H.
See Goetz, W. H. and Schaub, J. H.

Schick, H. L.
See John, R. R. and Schick, H. L.

Schieser, D. W.
See Tuck, L. D. and Schieser, D. W.

Schiffman, R. L.
The Use of Visco-Elastic Stress-Strain Laws in Soil Testing—Summary of Proceedings of *STP 254*, 1335.

Schlabach, Tom D.
The Temperature Dependence of Electrical Resistivity of Laminated Thermoset Materials. Published in ASTM BULLETIN, No. 238, May, 33 (TP 101) (*R*).

Schmidt, F. A.
See Carlson, O. N., Bare, D. W., Gibson, E. D., and Schmidt, F. A.

Schmidt, R. J.
See Hein, T. C. and Schmidt, R. J.

Schniewind, Arno P.
An Improved and Semi-Automatic Method of Conducting the Standard Hardness Test for Timber. Published in ASTM BULLETIN, No. 236, Feb., 57 (TP 67).

Schriever, B. A.
Materials in the Aerospace Age. Published in ASTM BULLETIN, No. 242, Dec., 16.

Schuette, E. H.
Fatigue-Strength Comparisons of Forged Magnesium Alloys--Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 274*).

Schultz, Edith G.
See Brunauer, Stephen, Copeland, L. E., Kantro, D. L., Weise, C. H., and Schultz, Edith G.

Schultz, W. W.
Grain Boundary Segregation Studies by Activation—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 268*).

Schutz, R. J. and Van Hauter, A.
New Concrete Joint Sealant Testing Machine. Published in ASTM BULLETIN, No. 241, Oct., 42 (TP 206).

Schwartzberg, F. R.
See Holden, F. C., Jaffee, R. I., and Schwartzberg, F. R.

Schwendiman, L. C., Brown, R. E., Honstead, J. F., Linderoth, C. E., and Pearce, D. W.
Disposal of Industrial Radioactive Waste Waters at Hanford—Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 273*).

Scott, A. H.
Activities of the International Electrotechnical Commission Dealing with Electrical Insulating Materials—Symposium on Electrical Insulating Materials—Summary of Proceedings of Third Pacific Area Meeting, 41 (*publication pending*).

Scott, R. K.
Introduction—Summary of Proceedings of *STP 256*, 1336.
Summary—Summary of Proceedings of *STP 256*, 1336.

Scott, Robert G.
The Structure of Synthetic Fibers—Summary of Proceedings of *STP 257*, 1074.

Scribner, B. F.
Introductory Remarks—Summary of Proceedings of *STP 259*, 1338.

Scribner, B. F. and Meggers, W. F.
Index to the Literature on Spectrochemical Analysis (Part IV, 1951–1955) (*issued as STP 41-D*).

Seebold, R. E.
See Birks, L. S. and Seebold, R. E.

Seed, H. B. and Fead, J. W. N.
Apparatus for Repeated Load Tests on Soils—Summary of Proceedings of *STP 254*, 1335.

Sell, R.
See Sachs, G., Sell, R., and Brown, Jr., W. F.

Semchyshen, Marion
Discussion, 984.

Semchyshen, M. and Barr, R. Q.
Mechanical Properties of Molybdenum and Molybdenum-Base Alloy Sheet—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 272*).

Sereda, P. J.
Measurement of Surface Moisture. Published in ASTM BULLETIN, No. 238, May, 61 (TP 129). Second Progress Report.

Sessler, John G.
Discussion, 874, 1014.

Shahinian, Paul
Discussion, 984.

Shank, Maurice E.
Summary of Survey on Projected Degree in Materials Engineering and Related Topics—Summary of Proceedings of *STP 263*, 1330 (*not to be published*).

Shapiro, J. J.
See Winslow, N. M. and Shapiro, J. J.

Sharpe, R. Q. and Henrikson, K. G.
Are Hydraulics Holding Their Own?—Symposium on Hydraulic Fluids (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 267*).

Sheeler, J. B.
See Davidson, D. T., Noguera, G., and Sheeler, J. B.

Shepherd, B. F.
Discussion, 1278.

Sheppard, H. R. and Ginsburg, H.
Should Statistical Methods Be Used to Prepare Materials Specifications. Published in ASTM BULLETIN, No. 236, Feb., 31 (TP 41).

Sherman, Morton
Discussion. Published in ASTM BULLETIN, No. 241, Oct., 25 (TP 189).

Shilton, R.
See Forbes, F. W. and Shilton, R.

Shobert, Erle I., II
Cooperation on Research Problems, ACR notes. Published in ASTM Bulletin, No. 239, July, 50.

Shoolery, J. N.
Nuclear Magnetic Resonance Spectroscopy—Symposium on Spectroscopy (Session IV)—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 269*).

Shubert, F. L.
See Goodwin, J. G., Rubenstein, L. S., and Shubert, F. L.

Shuttleworth, C. G.
Discussion, Published in ASTM Bulletin, No. 243, Jan., 1960, 51 (TP 9).

Siebert, C. A.
See Mikus, E. B. and Siebert, C. A.

Siegman, A. E.
Maser Applications and Traveling-Wave Techniques for Magnetic Resonance Spectroscopy—Symposium on Spectroscopy (Session IV)—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 269*).

Silver, I., Erickson, P. W., and Perry, H. A.
Problems in Evaluating Reinforcements—Symposium on Reinforced Plastics—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 279*).

Simmons, W. F., Sirois, B. J., Williams, D. N., and Jaffee, R. I.
Properties of 70-30 Copper-Nickel Alloy at Temperatures Ranging up to 1050 F, 1035 (*R*). Disc., 1046.

Simon, R. C.
Water Permeability Studies on Latex and Latex Paint Film—Session on Paints and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).

Simpson, W. C., Miles, T. K., and Griffin, R. L.
Correlation of Microfilm Durability Tests with Field Hardening Observed on Zaca-Wigmore Experimental Project—Symposium on Road and Paving Materials (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 277*).

Sipple, H. E.
See Francis, C. E. and Sipple, H. E.

Sirois, B. J.
See Simmons, W. F., Sirois, B. J., Williams, D. N., and Jaffee, R. I.

Sisco, Frank T.
Further Thoughts of Making Engineering Literature Available, ACR notes. Published in ASTM Bulletin, No. 240, Sept., 30.

Siviter, J. H., Jr.
Photographic Technique of Surface Temperature Measurement—Symposium on Thermal Ablation (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 279*).

Skidmore, Howard C.
Discussion, 1259.

Skog, J. B.
Results of Cooperative Test Series on Asphalts from the Zaca-Wigmore Experimental Project—Symposium on Road and Paving Materials (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 277*).
See Hveem, F. N., Zube, Ernst, and Skog, J. B.

Skupien, L. M.
See Alger, J. V. and Skupien, L. M.

Small, Louis
Discussion, 1275.

Smith C. R.
Designing Fatigue Resistant Structures—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 274*).

Smith, J. V.
Correlation of Elemental Analysis and Phase Identification as Viewed by a Mineralogist—Summary of Proceedings of *STP 256*, 1336.

Smith T. G.
New Developments in High Performance Fluids for Military Aircraft and Commercial Industrial Applications—Symposium on Hydraulic Fluids (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 267*).

Smith, Walter J.
A Promising Trend in Student Employment, ACR notes. Published in ASTM Bulletin, No. 241, Oct., 21.

Snodgrass, J. B.
Testing Models of Large Wood Roof Beam—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 275*).

Snyder, M. J.
Some Refractory Uranium Compounds—Preparation and Properties—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 276*).

Soderstrum, W. K.
See Johnson, P. V. and Soderstrum, W. K.

Solvason, K. R.
See Wilson, A. G., Solvason, K. R., and Novak, E. S.

Soroka, I.
Water-Retardant Qualities of External Coatings. Published in ASTM BULLETIN, No. 240, Sept., 40 (TP 170) (*R*).

Sosin, A.
Atomistic Interpretation of Radiation Effects in Metals—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 276*).

Sowers, George F.
Introduction—Summary of Proceedings of *STP 254*, 1335.

Sowers, G. F., Vesic, A., and Grandolfi, M.
Penetration Tests for Liquid Limit—Summary of Proceedings of *STP 254*, 1335.

Sparks, S. W.
See Brown, A. B. and Sparks, S. W.

Speight, F. Y.
Application of Statistics. Published in ASTM BULLETIN, No. 236, Feb., 29 (TP 39).

Spengeman, W. F. and Wormald, I.
Lightfastness Testing of Pigment Colors—Session on Paints and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).

Spinna, R. A.
See Morris, M. D., Ulp, R. B., and Spinna, R. A.

Squires, Frank
From Certainty to Statistics—Statistics Luncheon Address—Summary of Proceedings Third Pacific Area Meeting, 37. Published in ASTM BULLETIN, No. 243, Jan., 1960, p. 27.

Srawley, J. E.
Discussion, 872.

Standing, S. A. and Biondi, F. J.
Electron-Tube and Semiconductor Materials (Committee F-1 on Materials for Electron Tubes and Semiconductor Devices—Symposium on Materials in the Electronics Industry—Summary of Proceedings of Third Pacific Area Meeting, 43 (*not to be published*).

Stang, John H., Goldthwaite, William H., and Dunnington, Bruce W.
Design Studies of Capsules for High-Temperature Irradiations. Published in ASTM BULLETIN, No. 242, Dec., 54 (TP 214).

Steele, W. C.
See Wilson, M. K. and Steele, W. C.

Steidel, Jr., Robert F.
See Austin, Arthur L. and Steidel, Robert F., Jr.

Stein, Peter
Discussion. Published in ASTM BULLETIN, No. 239, July, 79 (TP 159).

Steiner, J. E.
See Kramer, K. H. and Steiner, J. E.

Stern, Milton and Weisert, Edward D.
Experimental Observations on the Relation Between Polarization Resistance and Corrosion Rate, 1280 (*R*). Disc., 1291.

Steurer, W. H.
Principles and Capabilities of Ablation—Symposium on Thermal Ablation (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 279*).

Stieda, C. K. A.
See Longworth, Jack and Stieda, C. K. A.

Stiehl, G. L., Jr.
Radiation Effects Problems in Unmanned Nuclear Propelled Vehicles—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 276*).

Stoll, P. A.
Electron Microscopy of Tin Plate—Summary of Proceedings of *STP 262*, 1072.

Stover, H. E.
See Dockweiler, E. V. and Stover, H. E.

Strait, L. A. and Hrenoff, M. K.
A Technique for Preparing Nonbrittle Solid Organic Samples for Infrared Analysis—Symposium on Spectroscopy (Session V)—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 269*).

Strella, S.
An Improved High-Speed Tension Tester. Published in ASTM BULLETIN, No. 236, Feb., 59 (TP 69). (*Technical Notes.*)

Sullivan, J. D.
See Engdahl, R. B. and Sullivan, J. D.

Sunderman, D. N.
The Radiochemical Determination of Uranium Burnup—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 276*).
See Howes, J. E., Jr., Brown, C. T., Sunderman, D. N., Pobereskin, M., and Schall, P.

Svendsen, Sven D. and Wingen, Robert
Norwegian Test Methods for Wind and Rain Penetration Through Windows—Summary of Proceedings of *STP 251*, 1337.

Swayze, Myron A.
Discussion, 1107.

Swenson, E. G.
See Thorvaldson, T. and Swenson, E. G.

Szirmae, A.
See Fisher, R. M. and Szirmae, A.

T

Taimuty, S. I.
Recent Developments in Gamma Dosimetry—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 276*).

Tavernelli, J. F. and Coffin, L. F., Jr.
Density Measurements on Strain-Cycled 2S Aluminum, 952.

Taylor, K. M.
See Wisnyi, L. G. and Taylor, K. M.

Taylor, K. M. and McMurtry, C. H.
Silicon Carbide-Clad-Graphite Matrix Fuel Elements—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 276*).

Taylor, W. H.
See Moorehead, D. R. and Taylor, W. H.

Thiers, R. E.
Flame Photometry from Medical and Biological Standpoints—Symposium on Spectroscopy (Session III)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 269*).

Thomas, J. P.
Accelerated Aging Tests and Life Aging Properties of Aircraft Metal Adhesives. Published in ASTM BULLETIN, No. 235, Jan., 58 (TP 28).

Thompson, R. D.
See Ruh, E. L., Moran, J. J., and Thompson, R. D.

Thompson, W. E.
See Kelley, R. M., Blank, E. W., Thompson, W. E., and Fine, R.

Thorvaldson, T. and Swenson, E. G.
Detection of Lignosulfonate Retarder in Cement Suspensions and Pastes—Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 266*).

Tietz, T. E.
Mechanical Properties of a High-Purity Lead and a 0.058 per Cent Copper-Lead Alloy at Elevated Temperatures, 1052. Disc., 1071.
Discussion, 983.

Tisiani, G. F. and Samans, C. H.
Additional Studies with an All-Glass Multiple-Test Apparatus for Nitric Acid Testing of Stainless Steels. Published in ASTM BULLETIN, No. 238, May, 64 (TP 132). (*Technical Note.*)

Toensing, C. H.
Processing and Properties of Tungsten—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 272*).

Toolin, P. R.
Discussion. Published in ASTM BULLETIN, No. 235, Jan., 51 (TP 21).
Discussion, 748.

Townsend, J. R.
International Standards, ACR notes. Published in ASTM BULLETIN, No. 236, Feb., 15.

Tramposch, Herbert, Papirno, Ralph, and Gerard, George
A Rapid Scanning System for Recording Thermocouple Outputs. Published in ASTM BULLETIN, No. 238, May, 41 (TP 109). (*Technical Note.*)

Transtrum, L. G.
See Pack, M. R., Hill, A. C., and Transtrum, L. G.

Tremper, Bailey
Control of Gypsum in Portland Cement, 1101. Disc., 1107.

Trice, J. B.
Techniques for Measuring Reactor Neutron Spectra—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 276*).

Tripp, Verne W.
See Rollins, Mary L., deGruy, Ines V., Tripp, Verne W., and Moore, Anna T.

Trojan, Paul K., Flinn, R. A., and Reese, D. J.
Behavior of Ductile Cast Iron Under Compressive Stress, 922.

Trout, Delmar E.
Effect of Gage on Rockwell Hardness Testing of Brass Strip. Report of Committee B-5 (Appendix II), 212.

Trozzo, P. S.
See McCartney, R. F. and Trozzo, P. S.
Techniques for Studying Structure and Growth of Tin Oxide by Electron Microscopy—Summary of Proceedings of *STP 262*, 1072.

Trudeau, L. P.
Irradiation of Some Pressure Vessel Steels—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 276*).

Tschaeche, A. M.
Dosimetry Nomenclature in the United States—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 276*).

Tuck, L. D. and Schieser, D. W.
Electron Spin Resonance Spectra of Free Radicals Derived from Some Polynuclear Heteroaromatic Compounds—Symposium on Spectroscopy (Session IV)—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 269*).

Tuckerman, Arnold
Effect of Environmental Exposure on Adhesive Bonded Structures—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 270*).

Tuthill, L. H., Adams, R. F., and Hemme, J. M., Jr.
Observations in Testing and Use of Water-Reducing Retarders—Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 266*).

U

Ulp, R. B.
See Morris, M. D., Ulp, R. B., and Spinna, R. A.

Umbach, R. D.
See Levy, D. F., Fassnacht, F. E., Hibler, G. P., Umbach, R. D., and Gagle, D. W.

V

Vandenbelt, J. M.
Problems in Ultraviolet Absorption Spectroscopy—Symposium on Spectroscopy (Session III)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 269*).

VandenBerg, R. V.
See Best, George E., Hecker, J. G., Jr., McGrew, J. W., and VandenBerg, R. V.

Vanderwerp, H. L.
Discussion, 1119.

Van Gundy, D. A.
See McClintock, R. M., Van Gundy, D. A., and Kropschot, R. H.

Van Hauter, A.
See Schutz, R. J. and Van Hauter, A.

Van Valkenburg, H. E.
See Panian, F. C. and Van Valkenburg, H. E.

Vendeland, R.
Potential Uses of Closed Circuit Television for Product Inspection—Summary of Proceedings of *STP 258*, 1331.

Verdi, Adam
See Lehman, H. L. and Adam, Verdi

Vesic, A.
See Sowers, G. F., Vesic, A., and Grandolfi, M.

Vind, H. P. and Hochman, Harry
A Standard Procedure for Evaluating the Toxicity of Chemical Agents to the Marine Borer Limnoria Tripuntata—Symposium on Treated Wood for Marine Use—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 275*).

Vollick, C. A.
Effect of Water-Reducing Admixtures and Set-Retarding Admixtures on the Properties of Plastic Concrete—Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 266*).

Vollick, Charles
Discussion, 1215, 1229.

Von Weisenstein, W.
See Dunn, E. J., Cestaro, J. P., and Von Weisenstein, W.

Vreeland, R. H.
A Test Method for Sonic Fatigue—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 274*).

Vreeland, T., Jr.
Discussion. Published in ASTM BULLETIN, No. 236, Feb., 47 (TP 57).

W

Waddell, Joseph J.
Control of Bituminous Shoulder Construction for the Northern Illinois Toll Highway—Summary of Proceedings of *STP 252*, 1332.

Waite, C. F.
Bibliography of References on Electroless Nickel Plating—Summary of Proceedings of *STP 265*, 1073.

Wakeman, C. M. and Whiteneck, L. L.
Extending the Life of Wood Piles in Sea Water—Symposium on Treated Wood for Marine Use—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 275*).

Walker, D. E.

See Noland, R. A., Walker, D. E., and Hymes, L. C.

Walker, Eric A.

Education in Engineering: a Perspective—Education in Materials Luncheon Address—Summary of Proceedings of *STP 263*, 1330.

Why Don't Engineers Learn to Write? address, education, luncheon, 62nd Annual Meeting (excerpt). Published in ASTM BULLETIN, No. 240, Sept., 20.

Wall, L. A.

Electron Spin Resonance Studies of Free Radicals in Irradiated Materials—Symposium on Post-Irradiation Effects in Polymers—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 276*).

Wallace, G. B.

See Higginson, E. C. and Wallace, G. B.

Wallace, G. B. and Ore, E. L.

Structural and Lean Mass Concrete as Affected by Water-Reducing, Set-Retarding Agents—Symposium on Effect of Water-Reducing and Set-Retarding Admixture on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 266*).

Warden, W. B.

Discussion. Published in ASTM BULLETIN, No. 235, Jan., 36 (TP 6).

Warren, Donald

Nitric Hydrofluoric Acid Evaluation Test for Type 316 L Stainless Steel. Discussion, published in ASTM BULLETIN, No. 235, Jan., 44 (TP 14).

Warren, Glenn B.

Further Challenges of the Design Engineer to the Metals Engineer and Their Bearings on the Metals Engineer's Education—Summary of Proceedings of *STP 263*, 1330.

Waters, K. T.

Production Methods of Cold Working Joints Subjected to Fretting for Improvement of Fatigue Strength—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 274*).

Weber, W. G., Jr.

Measurement of Excess Hydrostatic Pressures in Soils—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 52—Summary of Proceedings of *STP 254*, 1335.

Weber, W. G., Jr. and Kleiman, W. F.

Moisture and Strength Variation in a Thick, Uniform Clay Layer—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 52—Summary of Proceedings of *STP 254*, 1335.

Wechter, E. J.

Discussion, 1089.

Weise, C. H.

See Brunauer, Stephen, Copeland, L. E., Kantro, D. L., Weise, C. H., and Schultz, Edith G.

Weisert, Edward D.

See Stern, Milton and Weisert, Edward D.

Weiss, V.

X-ray Diffraction Studies of Several 52100 Steels and Tool Steels (appendix I to Sachs Sell, and Brown paper), 655.

White, R. M.

See Rostler, F. S. and White, R. M.

Whiteneck, L. L.

See Wakeman, C. M. and Whiteneck, L. L.

Wilcox, L. V.

Effect of Industrial Wastes on Waters for Irrigation Use—Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 273*).

Williams, D. N.

See Simmons, W. F., Sirois, B. J., Williams, D. N., and Jaffee, R. I.

Wilson, A. G., Solvason, K. R., and Novak, E. S.

Evaluation of Factory-Sealed Double-Glazed Window Units—Summary of Proceedings of *STP 251*, 1337.

Wilson, M. K. and Steele, W. C.

Some Comments Concerning Quantitative Infrared Spectroscopy—Symposium on Spectroscopy (Session V)—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 269*).

Wing, Paul and Monego, C. J.

Thermal Insulation Measurements on Textiles—A Comparison of Two Methods. Published in ASTM BULLETIN, No. 241, Oct., 29 (TP 193).

Wingen, Robert

See Svendsen, Sven D. and Wingen, Robert

Winslow, N. M. and Shapiro, J. J.

An Instrument for the Measurement of Pore-Size Distribution by Mercury Penetration. Published in ASTM BULLETIN, No. 236, Feb., 39 (TP 49) (*R*).

Winter, R. M.

See Parker, F. G., Winter, R. M., and LaPorta, J. A.

Winterkorn, Hans F.
Discussion, 1088.

Wisnyi, L. G. and Taylor, K. M.
Fabrication and Evaluation of Urania-Alumina Fuel Elements and Boron Carbide Burnable Poisons—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 276*).

Wohlers, H. C., Kass, T. E., and Johnson, K. R.
Fluorescent Dyes as Airborne Tracer Materials—Symposium on Air Pollution Control—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 281*).

Wood, J. C. S.
See Allen, J. G. and Wood, J. C. S.

Wood, J. Eldridge
Discussion. Published in ASTM BULLETIN, No. 235, Jan., 36 (TP 6).

Wood, L. W.
See Erickson, E. C. O. and Wood, L. W.

Wood, Melvin F.
Industry's Viewpoint on Education in Materials—Summary of Proceedings of *STP 263* 1330.

Woods, Hubert
Discussion, 1086.

Woods, K. B.
Introduction—Summary of Proceedings of *STP 263*, 1330.
The Impact of Science on ASTM—The President's Address, ASTM 62nd Annual Meeting. Published in ASTM BULLETIN, No. 239, July, 9.

Wormald, I.
See Spengeman, W. F. and Wormald, I.

Wright, C. C.
Field Testing of Corrosion Inhibitors in Sea Water—Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 273*).

Wycoff, John C.
Suitability of Lightweight Aggregate for Bituminous Plant Mix. Published in ASTM BULLETIN, No. 235, Jan., 33 (TP 3). Disc., 36 (TP 6).

Y

Yaeger, L. L.
See Lappala, R. P., Yaeger, L. L., and Bjorksten, Johan

Youden, W. J.
Statistical Aspects of the Cement Testing Program, 1120.

Yukawa, S. and McMullin, J. G.
High-Stress, Low-Cycle Fatigue Properties of Notched Alloy Steel Specimens. Published in ASTM BULLETIN, No. 241, Oct., 38 (TP 202).

Z

Zemer, R. F.
Effect of Natural Environment on Sandwich Structure Fabricated Using AMS 3722 Paper Honeycomb Core—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 270*).

Zieler, H. W.
Performance Characteristics of Objectives, Eyepieces, and Illuminators for Microscopy—Summary of Proceedings of *STP 257*, 1074.

Zube, Ernst
See Hveem, F. N., Zube, Ernst, and Skog, J. B.

SUBJECT INDEX

VOLUME 59

A

- Abraders
  - sandpaper machine
    - for testing floor coverings (Gavan). Published in ASTM BULLETIN, No. 238, May, 44 (TP 112).
- Absorption Spectroscopy
  - Report of Committee E-13, 607.
- Acoustical Materials
  - Report of Committee C-20, 414.
- ACR Notes. Published in ASTM BULLETIN, No. 235, Jan., 25; No. 236, Feb., 15; No. 237, April, 29; No. 239, July, 50; No. 240, Sept., 30; No. 241, Oct., 21; No. 242, Dec., 43.
  - *See also* Research.
- Activated Carbon
  - organic contaminant adsorbent (Laskie). Published in ASTM BULLETIN, No. 241, Oct., 41 (TP 205).
- Adhesives
  - bonded structures
    - effect of environmental exposure (Tuckerman)—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 270*).
  - for aircraft metal structures (Thomas). Published in ASTM BULLETIN, No. 235, Jan., 58 (TP 28).
  - Report of Committee D-14, 500.
  - Symposium on Adhesion and Adhesives—Summary of Proceedings of Third Pacific Area Meeting, 52 (*issued as STP 271*):
    - ceramic
      - for stainless steel
        - preliminary evaluation (Gates and Lent).
    - metal bonds
      - in Germany
        - lap shear and creep testing (Hahn).
    - metal-to-metal
      - elevated temperature resistant
        - derived from organic-inorganic polymer systems (Janis).
    - surface chemistry (Muchnick).
  - *See also* Laminates.
- Admixtures
  - water-reducing and set-retarding
    - Sympsoium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 45, 47 (*issued as STP 266*):
      - calcium sulfate
        - action in portland cement pastes (Hansen).
      - effect influenced by portland cement composition (Polivka and Klein).
      - effect on properties
        - of hardened concrete (MacPherson and Fischer).
        - of plastic concrete (Vollick).
      - effect on structural and lean mass concrete (Wallace and Ore).
      - introduction and summary (Foster).
      - lignosulfonate retarder
        - detection in cement suspensions and pastes (Thorvaldson and Swenson).
      - producers papers on (Prior and Adams).
      - uses; specifications; research objectives (Mielenz).
      - water-reducers
        - field experience in ready mixed concrete (Howard).
        - testing and use (Tuthill, Adams, and Hemme).
- Advertising
  - importance to the technical man. Published in ASTM BULLETIN, No. 240, Sept., 57.
  - directory of ASTM advertisers. Published in ASTM BULLETIN, No. 240, Sept., 57.
- Aerospace Age
  - materials in (Schriever). Published in ASTM BULLETIN, No. 242, Dec., 16.
- Aggregates
  - lightweight
    - from bituminous plant mix (Wycoff). Published in ASTM BULLETIN, No. 235, Jan., 33 (TP 3). Disc., 36 (TP 6).
  - limestone
    - fluid-flow measurements (Dolch), 1204 (*R*). Disc., 1214.
  - mineral
    - degradation (Minor)—Symposium on Road and Paving Materials—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 277*).
    - test for production of plastic fines (Ekse and Morris)—Symposium on Road and Paving Materials—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 277*).
  - Report of Committee C-9, 387.

  - separation from lightweight material (Higginson and Wallace). Published in ASTM Bulletin, No. 243, Jan., 1960, p. 60 (TP 18).
- Aircraft and Missile Materials
  - metal adhesives
    - aging test (Thomas). Published in ASTM Bulletin, No. 235, Jan., 58 (TP 28).
  - stainless-steel honeycombs (Gilbraith). Published in ASTM Bulletin, No. 240, Sept., 51 (TP 181).
- Air Pollution
  - Symposium on Air Pollution Control—Summary of Proceedings of Third Pacific Area Meeting, 53, 54 (*to be published as STP 281*):
    - and chemical plant design
      - wind and weather summaries (Moore, Boscia and Lundeen).
    - fluorescent dyes as airborne tracer materials (Wohlers, Kass, and Johnson).
    - formaldehyde and methanol from combustion sources
      - colorimetric determination (Feldstein).
    - gaseous and particulate inorganic fluorides in the atmosphere (Pack, Hill and Transtrum).
    - implications of the Ram strati-charge engine—panel discussion (Heintz, Kadoch, Linville, and Linsky).
    - potential of California coastal climate (Robinson).
    - Ram strati-charge engine (Heintz and Kadoch).
- Alloy Steel
  - Report of Committee A-6, 156.
- Aluminum
  - adhesion bonded sandwich structures
    - ultrasonic inspection (Kammerer)—Symposium on Nondestructive Testing in the Missile Industry—Summary of Third Pacific Area Meeting, 41 (*to be published in STP 278*).
  - alloys
    - long-range segregation of uranium (Peckner). Published in ASTM Bulletin, No. 242, Dec., 51 (TP 211).
    - wrought
      - chromic acid anodizing characteristics (Best, Hecker, Jr., McGrew, and Vandenberg). Report of Committee B-7 (appendix II), 277 (*R*).
  - alloy specimens
    - variable amplitude fatigue tests (Hardrath and Naumann)—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 274*).
  - appearance measuring (Paulson). Published in ASTM Bulletin, No. 242, Dec., 33.
  - atmospheric exposure tests
    - ASTM test sites (Adams and Dougherty). Report of Committee B-7 (appendix III), 289.
  - cold-rolled sheet
    - crack propagation (Lipsitt, Forbes, and Baird), 734 (*R*). Disc., 748.
  - corrosion
    - Report of Advisory Committee on Corrosion, 333 (*R*).
  - fuel elements
    - U_3O_8-Al
      - fabrication by extrusion (Noland, Walker, and Hymes)—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 276*).
    - urania-alumina and boron carbide burnable poisons
      - fabrication and evaluation (Wisnyi and Taylor)—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 276*).
  - Report of Committee B-1, 163.
  - Report of Committee B-6, 262.
  - Report of Committee B-7, 263.
  - strain-cycled 2S
    - fatigue damage study
      - by density measurements (Tavernelli and Coffin, Jr.), 952.
  - tensile properties
    - at high rates of strain (Austin and Steidel), 1292 (*R*), Disc., 1307.
  - wrought alloys
    - chromic acid anodized coatings (Best, Hecker, Jr., McGrew, and VandenBerg). Report of Committee B-7 (appendix II) (*R*).
- American Standards Association
  - approves ASTM standards as American Standard. Published in ASTM Bulletin, No. 236, Feb., 14; No. 237, April, 29; No. 238, May, 17; No. 239, July, 51.
- Analytical Chemistry
  - expanding horizons (Gambrill). Published in ASTM Bulletin, No. 239, July, 45.
- Annual Meeting
  - Report of Board of Directors, 61.
  - Summary of Proceedings of Sixty-second Annual Meeting, 1.
  - *See also* Meetings.
- Anodizing
  - chromic acid coatings
    - on wrought aluminum alloys (Best, Hecker, Jr., McGrew, and VandenBerg). Report of Committee B-7 (appendix II), 277 (*R*).
  - coatings
    - Report of Committee B-7, 263.
- Antifreezes
  - *See* Engine Antifreezes.
- Antimony
  - spectrochemical analysis
    - suggested method
      - by the point-to-plane spark technique (Dunn, Cestaro, and Von Weisenstein) Report of Committee E-2 (appendix I), 573 (*issued as separate reprint*).
- Appearance
  - in aluminum surfaces (Paulson). Published in ASTM Bulletin, No. 242, Dec., 33.
  - Report of Committee E-12, 606.

Aromatic Hydrocarbons
  Report of Committee D-16, 505.
Asbestos
  cotton content
    referee method
      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XI), p. 812.
Asbestos-Cement Products
  Report of Committee C-17, 410.
Asbestos Materials
  for electrical insulation
    iron content rate (Nicodemus). Published in ASTM Bulletin, No. 237, April, 62 (TP 82). Disc., 67 (TP 87).
Askarels
  Report of Commiteee D-27, 550.
Asphalt
  coating grade
    weatherability
      effects of composition and processing (Mertens and Greenfeld)—Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 280*).
  Report of Committee D-4, 449.
  Report of Committee D-8, 466.
  roofing
    composition and rheology (Brown and Sparks)—Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 280*).
    manufacture and application (Haack)—Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 280*).
  Symposium on Road and Paving Materials—Summary of Proceedings of Third Pacific Area Meeting, 48, 49, 50 (*to be published in STP 277*):
    chemical composition
      influence on performance (Rostler and White).
    mixtures
      temperature effect on flexibility (Monismith).
    Symposium on the Zaca-Wigmore Experimental Test Road
      cooperative test series (Skog).
      correlation of durability tests with field hardening (Simpson, Miles, and Griffin).
      progress report (Hveem, Zube, and Skog).
  See also Bituminous Paving Materials.
ASTM
  and the critical data problem. Published in ASTM Bulletin, No. 236, Feb., 16.
  and impact of science, President's address (Woods). Published in ASTM Bulletin, No. 239, July, 9.
  awards of merit. Published in ASTM Bulletin, No. 239, July, 24.
  directory of advertisers. Published in ASTM Bulletin, No. 240, Sept., 57.
  finances—1958. Published in ASTM Bulletin, No. 236, Feb., 10.
  flammability
    ASTM test method improvements (Eysenbach and Gouza). Published in ASTM Bulletin, No. 242, Dec., 62 (TP 222).
  honorary members elected 1959. Published in ASTM Bulletin, No. 239, July, 23.
  industrial chemicals
    new committee (E-15). Published in ASTM Bulletin, No. 237, April, 48.
  in 1958. Published in ASTM Bulletin, No. 235, Jan., 5.
  materials and progress (LaQue). Published in ASTM Bulletin, No. 242, Dec., 20.
  national officers elected 1959. Published in ASTM Bulletin, No. 239, July, 20.
  new division on materials sciences. Published in ASTM Bulletin, No. 236, Feb., 5.
  property
    headquarters, additional acquisition. Published in ASTM Bulletin, No. 236, Feb., 9.
  publications 1959–1960. Published in ASTM Bulletin, No. 241, Oct., 5.
  reports file. Published in ASTM Bulletin, No. 241, Oct., 12.
  the Society's name. Report of the Board of Directors, 62. Published in ASTM Bulletin, No. 241, Oct., 12.
  test sites. Published in ASTM Bulletin, No. 236, Feb., 24.
  two new technical committees. Published in ASTM Bulletin, No. 237, April, 48.
Atmospheric Corrosion
  of test sites
    Report of Subgroup of Subcommittee VII of Committee B-3 (appendix II), 183.
  Report of Committee B-7, 263.
  Report of Subcommittee VI of Committee B-3 (appendix I), 176.
Atmospheric Exposure
  Advisory Committee on Corrosion. Published in ASTM Bulletin, No. 236, Feb., 24.
  aluminum and magnesium alloys
    ASTM test sites (Adam and Dougherty). Report of Committee B-7 (appendix III), 289.
  1958—of hardware specimens
    Report of Subcommittee XVI of Committee A-5, 133.
  Report of Committee A-5, 127.
Atterberg Limits
  Symposium. Summary of Proceedings of *STP 254*, 1334:
    correlation with geology of deep cores (Johnson and Morris).
    history and development of tests (Bauer).
    introduction (Sowers).
    liquid limit tests
      investigation of (Dawson).
      one-point
        mechanical method (Joslin and Davis).
        procedure (Eden).

      - penetration tests (Sowers, Vesic, and Grandolfi).
      - proposed universal standards (Morris, Ulp, and Spinna).
      - use of grooving tools (Mitchell).
    - plastic limit
      - cube and standard thread methods (Abdun-Nur).
- Awards
  - citations for quality of presentation. Published in ASTM BULLETIN, No. 239, July, 22.
  - committee D-12, J. C. Harris—1959 recipient. Published in ASTM BULLETIN, No. 236, Feb., 23.
  - Harold De Witt Smith, address (Hamburger). Published in ASTM BULLETIN, No. 238, May, 23.
  - honorary members. Published in ASTM BULLETIN, No. 239, July, 23.
  - of merit (ASTM). Published in ASTM BULLETIN, No. 239, July, 24.
  - Published in ASTM BULLETIN, No. 236, Feb., 23; No. 239, July, 24.
  - Report of the Board of Directors, 75.

B

- Beams
  - concrete
    - central and third-point loading
      - modulus of rupture (Raville and McCormick), 1230.
  - laminated wood
    - strength studies of scarf joints (Longworth and Stieda)—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 275*).
- Bending
  - notch
    - determination of energy release (Lubahn), 885 (*R*). Disc., 914.
- Benzene
  - thiophene in
    - results of cooperative program. Report of Committee D-16 (appendix III), 514.
- Beryllium
  - elevated temperature properties
    - effect of purity and manufacturing variables (Beaver, O'Rourke, and Hurd)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 272*).
  - oxide
    - for nuclear applications (Hove)—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 276*).
  - QMV
    - stress-rupture and creep properties (Hurd, O'Rourke, and Beaver)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 53 (*published in STP 272*).
  - Report of Committee B-2, 171.
- Bituminous Materials
  - highway construction (Armstrong). Published in ASTM BULLETIN, No. 241, Oct., 14.
  - Report of Committee D-4, 449.
  - Report of Committee D-8, 466.
  - Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 52, 54 (*to be issued as STP 280*):
    - asphalt
      - coating grade
        - weatherability
          - effects of composition and processing (Mertens and Greenfeld).
      - roofing
        - composition and rheology (Brown and Sparks).
        - manufacture and application (Haack).
    - glass fiber felt roofing products (Hayes).
    - grouts, mastics and cold applied cements (Butterfield and Haney).
    - pitch
      - coal tar flat (Fair, Jr. and Chamberlain).
      - industrial (Pittman and Donegan).
- Bituminous Mixtures
  - lightweight aggregates (Wycoff). Published in ASTM BULLETIN, No. 235, Jan., 33 (TP 3). Disc., 36 (TP 6).
- Bituminous Paving Materials
  - Symposium on Methods of Test for Design of Bituminous Paving Mixtures. Symposium on Practical and Statistical Significance of Tests and Properties of Bituminous Binders. Papers on Road and Paving Materials. Summary of Proceedings of *STP 252* (Bituminous Paving Materials), 1332:
    - binders
      - significance of tests and properties
        - ASTM tests and specifications
          - evolution (Abson).
          - present, precision of (Brown).
          - possibilities for extension and improvement (Fink).
        - fundamental statistical concepts (Irick).
        - practical (McLeod).
    - design
      - methods of test
        - application of Marshall method to hot-mix (Lehmann and Adam).
        - correlation of results
          - Hveem stabilometer and cohesiometer, kneading compactor densities, service records (Minor).
        - general factors (Rader).
        - gyratory testing machine for hot-mix (McRae and Foster).
        - immersion-compression test (Goode).
        - highway shoulder construction (Ill.) (Waddell).
        - triaxial testing (Goetz and Schaub).
        - void requirements for dense-graded mixtures (McLeod)
    - introduction (Fink)
    - road and paving materials

- Bituminous Paving Materials (*continued*)
  - density changes in asphalt core samples (Hein and Schmidt).
  - refinery control of cutback asphalt
    - fundamental viscosity *versus* Sayboldt Furol viscosity (Levy, Fassnacht, Hibler, Umbach and Gagle).
- Board of Directors
  - auditor's report (appendix I), 81.
  - Report of Administrative Committee on District Activities (appendix IV), 106.
  - Report of Administrative Committee on Papers and Publications (appendix III), 102.
  - Report of Administrative Committee on Research (appendix V), 107.
  - Report of Administrative Committee on Simulated Service Testing (appendix VI), 109.
  - Report of Administrative Committee on Standards (appendix II), 95.
  - Report of Board of Directors, 61.
- Boiler Feedwater
  - Report of Committee D-19, 523.
- Brass
  - cartridge
    - annealing behavior (Pulsifer), 930 (*R*).
  - Report of Committee B-5, 205.
  - Rockwell hardness test
    - effect of thickness (Trout). Report of Committee B-5 (appendix II), 212.
  - strip
    - hardness test
      - effect of gage (Trout). Report of Committee B-5 (appendix II), 212.
- Brass Strip
  - Rockwell hardness testing
    - effect of gage (Trout). Report of Committee B-5 (appendix II), 212.
- Brick
  - rate of moisture absorption (Hodgins). Published in ASTM Bulletin, No. 241, Oct., 27 (TP 191).
  - reinforced grouted
    - values in diagonal tension and shear (Kelch)—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 275*).
  - Report of Committee C-15, 402.
  - *See also* Fireclay Brick *and* Moisture.
- Bronze
  - Report of Committee B-5, 205.
  - tensile properties
    - effect of low-temperature (McClintock, Van Gundy, and Kropschot). Published in ASTM Bulletin, No. 240, Sept., 47 (TP 177).
- Building Constructions
  - Report of Committee E-6, 591.
  - Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 275*):
    - diaphragms
      - adequacy in resisting horizontal forces (Barnes).
      - large, testing of (Cousineau).
    - hollow concrete masonry wall panels
      - values in diagonal tension and shear (Kelch) (*publication pending*).
    - introduction (Legget).
    - large wood roof beam
      - testing models (Snodgrass).
    - light gage steel building
      - lateral shear tests (Holstein) (*publication pending*).
    - reinforced grouted brick masonry
      - values in diagonal tension and shear (Kelch) (*publication pending*).
    - yard lumber of common grade
      - determining useful strength (May).
  - *See also* Sandwich Constructions.
  - Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 46, 48 (*to be published in STP 275*).
    - laminated beams
      - strength studies of scarf joints (Longworth and Stieda).
    - quality control and inspection (Hanrahan).
    - residential in California
      - termite control (Ebeling).
    - school in California
      - renaissance of wood (Maag).
    - structural design
      - deflection limitations (Eby).
      - need for special engineering education (Glaze)
    - western woods
      - screw-holding ability: effects of test variables (Johnson).
    - wood-pole (Keeney).
      - strength and related properties (Erickson and Wood).
- Building Materials
  - codes and standards (Legget). Published in ASTM Bulletin, No. 237, April, 36.
  - masonry
    - Symposium on Masonry Materials. Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*):
      - concrete block
        - structural bond of mortar and grout (Copeland).
      - lightweight clay units properties
        - relation of current ASTM specifications (Johnson and Soderstrum).
      - mortar for efflorescence (Rogers).
      - reinforced construction (Mackintosh).
      - reinforced grouted brick
        - mortar and grout field test specimens (Kelch).
  - sealants
    - for concrete joints
      - testing machine (Schutz and Van Hauter). Published in ASTM Bulletin, No. 241, Oct., 42 (TP 206).
    - proposed specification
      - for building trade
        - polysulfides. Published in ASTM Bulletin, No. 242, Dec., 46.
  - *See also* Beams, Brick, Cement, Concrete, Glass, Mortar, Plastics, Sandwich Construction, Timber, Waterproofing, Window Assemblies, *and* Wood.

Bulk Modulus
  measurement of
    in hydraulic fluids (Peeler and Green). Published in ASTM BULLETIN, No. 235, Jan., 51 (TP 21) (*R*).
  proposed definitions. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix IV), p. 1138.
  See also Hydraulic Fluids *and* Proposed Definitions.

C

Cables
  high voltage insulation
    Symposium. Summary of Proceedings of *STP 253*, 1340:
      oils
        utility viewpoint (Gates and Gillette).
        why and how of tests (Blodgett).
      paper (Delmar).
  Report of Committee B-1, 163.
Calcium Oxide
  in hydrated lime (Moorehead and Taylor). Published in ASTM BULLETIN, No. 236, Feb., 45 (TP 55).
Capsules
  for high-temperature irradiations (Stang, Goldthwaite, and Dunnington). Published in ASTM BULLETIN, No. 242, Dec., 54 (TP 214).
Carbon Black
  Report of Committee D-24, 546.
Casein
  Report of Committee D-25, 548.
Cast Iron
  ductile
    compressive stress (Trojan, Flinn, and Reese), 922.
  Report of Committee A-3, 124.
Cathodes
  Report of Committee F-1, 628.
Causticaire Scale
  for estimating maturity and weight of cotton fibers
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XIV), p. 830.
Cellulose
  Report of Committee D-23, 544.
Cellulose Finishes
  on textiles
    identification
      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix VII), p. 803.
Cement
  Cement Reference Laboratory
    origin, purpose, development (Dise). Report of Committee C-1 (appendix II), 369.
  granular soil-cement mixtures for base-courses (Abrams). Summary of Proceedings of *STP 254*, 1335.
  highway construction (Armstrong). Published in ASTM BULLETIN, No. 241, Oct., 14.
  interlaboratory tests
    statistical aspects (Youden), 1120
    statistical evaluation (Crandall and Blaine), 1129
  portland
    acetate processed (Kester), 1111. Disc., 1118.
    control of gypsum (Tremper), 1101. Disc., 1107.
    control of SO_3
      short-time tests of mortars (Report of Working Committee on SO_3 Content). Report of Committee C-1 (appendix I), 349.
    determination of four major phases
      X-ray quantitative analysis (Brunauer, Copeland, Kantro, Weise, and Schultz), 1091.
    determination of magnesium oxide
      by radiometric method (Howes, Jr., Brown, Sunderman, Pobereskin, and Schall)—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 268*).
    magnesium oxide in (Berman). Published in ASTM BULLETIN, No. 237, April, 51 (TP 71).
    release of alkalies (Hansen). Published in ASTM BULLETIN, No. 236, Feb., 35 (TP 45).
    SO_3 content. Report of Committee C-1 (appendix I), 349.
    X-ray spectrography
      quantitative light element analysis Fe to Mg (Curley)—Symposium on Spectroscopy (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 269*).
  portland blast-furnace slag
    ignition loss (Chaiken). Published in ASTM BULLETIN, No. 238, May, 53 (TP 121).
  proposed method of test
    for determining cement content in fresh concrete. Published in ASTM BULLETIN, No. 239, July, 48.
  Report of Committee C-1, 345.
  thermal insulation
    Report of Committee C-16, 406.
  water-reducing and set-retarding admixtures
    Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 45, 47 (*published in STP 266*):
      introduction and summary (Foster).
      portland
        effect of compositions on admixtures (Polivka and Klein).
        pastes
          action of calcium sulfate (Hansen).
      producers papers on (Prior and Adams).
      suspensions and pastes
        detection of lignosulfate retarder (Thorvaldson and Swenson).

Cement (*continued*)
  (Candela)—Cement and Concrete Industry Luncheon—Summary of Proceedings of Third Pacific Area Meeting, 38 (*not to be published*).
  See also Admixtures *and* Reference Method.
Cemented Carbides
  Report of Committee B-9, 330.
  Rockwell hardness test (Caugherty, Oatman, and Reen), 1261. Disc., 1275.
Cement Reference Laboratory
  (Dise). Report of Committee C-1 (appendix II), 369.
Ceramic
  adhesives for stainless steel (Gates and Lent)—Symposium on Adhesion and Adhesives—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 271*).
  in nuclear energy
    Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 39, 40 (*to be published in STP 276*).
      beryllium oxide (Hove).
      forming of particles
        for fuels, control and burnable poison dispersion components (Ploetz and Muccigrosso).
      fuel elements
        dry pressing to close tolerances (Hartwig).
        graphite matrix (Riley).
          silicon carbide clad (Taylor and McMurtry).
        U_3O_8-Al
          fabrication by extrusion (Noland, Walker, and Hymes).
        urania-alumina and boron carbide burnable poisons
          fabrication and evaluation (Wisnyi and Taylor).
      refractory uranium compounds—preparation and properties (Snyder).
  Report of Committee F-1, 628.
Ceramic Coatings
  Report of Committee C-22, 418.
Ceramic Whitewares
  Report of Committee C-21, 416.
Chemical Analysis
  photometric methods
    infrared
      proposed recommended practice (*issued as separate ASTM reprint*).
    ultraviolet
      proposed recommended practice (*issued as separate ASTM reprint*).
  Report of Committee E-2, 569.
  Report of Committee E-3, 581.
  See also Analytical Chemistry.
Chemical-Resistant Mortars
  Report of Committee C-3, 378.
Chemical-Resistant Units
  Report of Committee C-15, 402.
"Chimmey" Test Method
  for water-retardant coatings (Soroka). Published in ASTM Bulletin, No. 240, Sept., 40 (TP 170) (*R*).
"Chromanalyzer" Apparatus
  and apparatus in method D 1319
    cooperative comparative data. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix V), p. 1140.
  for hydrocarbon analysis of olefinic gasolines (Allen and Wood). Published in ASTM Bulletin, No. 238, May, 49 (TP 117).
Chromatography
  analysis of sodium tripolyphosphate (Kolloff). Published in ASTM Bulletin, No. 237, April, 74 (TP 94) (*R*).
  column
    for analysis of creosote (Drisko and Hochman)—Symposium on Treated Wood for Marine Use—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 275*).
Chromium
  deoxidation and denitrification
    effect on low temperature properties (Levingston and Grant)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 272*).
Clay
  and organic solids
    strain-rate behavior (Burmister). Summary of Proceedings of *STP 254*, 1335.
  calcium-45-tagged
    as a detergency test soil (Hensley and Inks)—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 268*).
  lightweight masonry units properties
    relation of current ASTM specifications (Johnson and Soderstrum)—Symposium on Masonry Materials—Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*).
  sensitive
    determination of preconsolidation pressure (Hamilton and Crawford). Summary of Proceedings of *STP 254*, 1335.
    rate of strain on effective stresses (Crawford). Summary of Proceedings of *STP 254*, 1335.
  thick uniform layer
    moisture and strength variation (Weber, Jr. and Kleiman)—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 52—Summary of Proceedings of *STP 254*), 1335.
  time effects on consolidation (Leonards and Ramiah). Summary of Proceedings of *STP 254*, 1335.
  See also Filter Block, Clay.
Clay Pipe
  Report of Committee C-4, 380.
Coal
  Report of Committee D-5, 454.
Coal Chemicals
  Report of Committee D-16, 505.

- Coatings
  - chromic acid anodized
    - on wrought aluminum alloys (Best, Hecker, Jr., McGrew, and VandenBerg). Report of Committee B-7 (appendix II), 277 (*R*).
  - electrodeposited
    - Report of Committee B-8, 315.
    - standardizing preparation
      - corrosion (Graham and Pinkerton). Report of Committee B-8 (appendix), 317 (*R*).
    - supplementary coatings
      - Report of Committee B-8, 315.
  - on fabrics
    - radioisotope methods of testing uniformity (Foster)—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 268*).
  - organic
    - thickness measurement
      - by infrared interferometry (Brightwell). Published in ASTM Bulletin, No. 237, April, 67 (TP 87).
  - paint
    - on steels (Copson and Larrabee). Published in ASTM Bulletin, No. 242, Dec., 58 (TP 228) (*R*).
  - Report of Committee B-7, 263.
  - very thin vacuum evaporated metal films
    - tracer techniques (Preuss)—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 268*).
  - water-retardant
    - "Chimney" test method (Soroka). Published in ASTM Bulletin, No. 240, Sept., 40 (TP 170) (*R*).
  - wet-abrasion resistance test
    - for coating films (Diamond). Published in ASTM Bulletin, No. 236, Feb., 48 (TP 58) (*R*).
  - *See also* Filters.
- Coating Materials
  - wet-abrasion resistance test (Diamond). Published in ASTM Bulletin, No. 236, Feb., 48 (TP 58) (*R*).
- Coke
  - Report of Committee D-5, 454.
- Color
  - Report of Committee E-12, 606.
- Columbium (Niobium)
  - fabrication (McCullough)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 272*).
  - Report of Committee B-2, 171.
  - workability and mechanical properties
    - effect of oxygen and nitrogen (Begley and France)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 272*).
- Committee Work
  - and fundamental research (Harris). Published in ASTM Bulletin, No. 238, May, 18.
- Communication
  - plain language (Mowbray). Published in ASTM Bulletin, No. 241, Oct., 18.
- Compression
  - concrete
    - effect of length-diameter ratio (Kesler), 1216. Disc., 1229.
  - concrete cubes
    - influence of direction of loading (Neville). Published in ASTM Bulletin, No. 239, July, 63 (TP 143).
  - ductile cast iron (Trojan, Flinn, and Reese), 922.
  - metals
    - at constant true strain rates (Hockett), 1309. Disc., 1317.
  - properties of steels
    - for aircraft bearings (Sachs, Sell, and Brown, Jr.) 635 (*R*). Disc., 658.
  - wood
    - at elevated temperatures (Ingberg), 1320.
- Concrete
  - alkali-aggregate reaction
    - mineral admixtures preventing expansion (Pepper and Mather), 1178 (*R*). Disc., 1202.
  - beams
    - central and third-point loading
      - modulus of rupture (Raville and McCormick), 1230.
  - cement content of freshly mixed
    - proposed method of test. Published in ASTM Bulletin, No. 239, July, 48.
  - compression
    - effect of length to diameter ratio (Kesler). (Committee C-9, Subcommittee IIIa investigation), 1216. Disc., 1229.
    - influence of direction of loading (Neville). Published in ASTM Bulletin, No. 239, July, 63 (TP 143).
  - highway (Cahn and Mackay, Jr.). Published in ASTM Bulletin, No. 235, Jan., 37 (TP 7). Disc., 41 (TP 11).
  - highway construction (Armstrong). Published in ASTM Bulletin, 241, Oct., 14.
  - hollow masonry wall panels
    - values in diagonal tension and shear (Kelch)—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*publication pending*).
  - in-place strength test (Benjamin and Ratliff, Jr.). Published in ASTM Bulletin, No. 241, Oct., 23 (TP 187). Disc., 25 (TP 189).
  - joint sealant materials (Schutz and Van Hauter). Published in ASTM Bulletin, No. 241, Oct., 42 (TP 206).
  - pavement design and evaluation
    - repetitive plate load tests (Gordon)—Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 254*).
  - Report of Committee C-9, 387.

Concrete (*continued*)
  water-reducing and set-retarding admixtures
    Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete—Summary of Proceedings of Third Pacific Area Meeting, 45, 47 (*published in STP 266*):
      hardened, effect on properties (MacPherson and Fischer).
      influence of portland cement composition (Polivka and Klein).
      introduction and summary (Foster).
      plastic effect on properties (Vollick).
      producers papers on (Prior and Adams).
      ready mixed, field experience (Howard).
      structural and lean mass, effect on (Wallace and Ore).
      use, observations in testing (Adams and Hemme).
      uses; specifications; research objectives (Mielenz).
  (Candela)—Cement and Concrete Industry Luncheon—Summary of Proceedings of Third Pacific Area Meeting, 38 (*not to be published*).
  See also Admixtures
Concrete Masonry Units
  Report of Committee C-15, 402.
Concrete Pipe
  Report of Committee C-13, 396.
Conditioning
  psychrometric table for relative humidity
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix II), p. 778 (*also in Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 567 (1931)).
Conductors
  Report of Committee B-1, 163.
Containers
  Report of Committee D-10, 477.
Cooperative Activities
  Report of Board of Directors, 79.
Copper
  alloys
    creep and rupture properties at 550 to 650 F (Kinsey), 940.
  magnetic properties (Parker, Winter, and LaPorta). Report of Committee B-5 (appendix III), 230 (*R*).
  Report of Committee B-2, 171.
  Report of Committee B-5, 205.
  70-30 copper-nickel alloy
    properties at temperatures up to 1050 F (Simmons, Sirois, Williams, and Jaffee), 1035. Disc., 1046.
  tensile properties
    effect of low-temperature (McClintock, Van Gundy, and Kropschot). Published in ASTM BULLETIN, No. 240, Sept., 47 (TP 177).
  See also Lead.
Copper Wire
  Report of Committee B-1, 163.
Corrosion
  ASTM atmospheric exposure sites (Advisory Committee on Corrosion). Published in ASTM BULLETIN, No. 236, Feb., 24.
  electrochemical measurements
    by polarization resistance (Stern and Weisert), 1280 (*R*). Disc., 1291.
  hardware, 1958 test
    Report of Subcommittee XVI of Committee A-5 (appendix) 133.
  measurement of surface moisture (Sereda). Published in ASTM BULLETIN, No. 238, May, 61 (TP 129).
  protection by metal preservatives
    proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix VII), p. 1146.
  Report of Advisory Committee on Corrosion, 333 (*R*).
  Report of Committee A-5, 127.
  Report of Committee A-10, 158.
  Report of Committee B-3, 174.
  Report of Committee B-6, 262.
  Report on Stress-Corrosion Cracking of Austenitic Chromium-Nickel Stainless Steels. *Issued as STP 264:*
    bibliography
    case history
      United Kingdom
      U. S. A.
    classification
      of corrosion conditions (Bloom).
      of materials and stress conditions (Brooks and Holmberg).
    fractures and associated conditions (Denhard).
    general information (Dana, Jr., and Friend).
    introductory summary (LaQue).
    mechanism (Harwood).
    research
      survey of activities (Dana, Jr. and Friend).
      steps for overcoming (Bloom).
  stainless steel
    nitric acid all-glass multiple-test apparatus (Tisinai and Samans). Published in ASTM BULLETIN, No. 238, May, 64 (TP 132).
  testing
    electrodeposits on panels
      standardizing the preparation (Graham and Pinkerton). Report of Committee B-8 (appendix), 317 (*R*).
    program—1957
      Report of Subcommittee VI of Committee B-3 (appendix I), 176.
Corrosion Resistance
  Report of Committee A-10, 158.
Corrosiveness of Test Sites
  Report of Committee B-3, 174.
  Report of Subgroup of Subcommittee VII of Committee B-3 (appendix II), 183.
Cotton and Cotton Fabrics
  basic properties of fibers
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix I), p. 777.
Cotton and Cotton Products
  causticaire method
    for estimating maturity and weight per unit length of fibers

      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials, (appendix XIV), p. 830.
  determining average quality
    specification of number of tests
      proposed recommended practice. Published in 1959 Compilation of ASTM Standards on Textile Materials (appendix IV), p. 791.
  finishes, identification
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix VII), p. 803.
  in asbestos textiles
    referee method
      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XI), p. 812.
  length and length distribution of fibers
    by short method array
      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XIII), p. 824.
  strength of fibers
    by finite gage length (flat bundle method)
      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XII), p. 817.
Crack Propagation
  fatigue
    method for predicting rate of (McEvily, Jr. and Illg)—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 274*).
Crease Test
  for textile fabrics
    by roller pressure apparatus
      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix VIII), p. 806.
Creep
  anelastic
    effect of temperature (Lubahn). Published in ASTM Bulletin, No. 239, July, 61 (TP 141). Disc., No. 243, Jan., 1960, 54 (TP 12).
  effect of environment
    on ferritic and austenitic stainless steels (Garofalo), 973 (*R*). Disc., 983.
  metals
    literature surveys on influence of stress concentrations at elevated temperatures and effects of nonsteady load and temperature conditions (Brown, Jr., Manson, Sachs, and Sessler) (*issued as STP 260*).
  properties of copper-base alloys at 550 to 650 F (Kinsey), 940.
  304, 316, 321 austenitic stainless steel
    notched and unnotched specimens (Garofalo), 957 (*R*).
Creosote
  analysis of
    by use of column chromatography (Drisko and Hochman)—Symposium on Treated Wood for Marine Use—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 275*).
Critical Data
  and ASTM. Published in ASTM Bulletin, No. 236, Feb., 16.
Cufenloy 30
  copper-nickel alloy
    physical and mechanical properties at elevated temperatures (Simmons, Sirois, Williams, and Jaffee), 1035 (*R*). Disc., 1046.
Cryogenics
  hydrogen bubble chambers
    engineering of (Birmingham, Chelton, Mann, and Hernandez). Published in ASTM Bulletin, No. 240, Sept., 34 (TP 164) (*R*).
Curing
  vulcanizable materials
    time rates from Mooney test (Juve). Published in ASTM Bulletin, No. 239, July, 71 (TP 151).

D

Data Storage and Retrieval
  critical. Published in ASTM Bulletin, No. 236, Feb., 16.
  making engineering literature available, ACR notes (Sisco). Published in ASTM Bulletin, No. 240, Sept., 30.
Demulsibility
  of high viscosity oils (Brennan). Published in ASTM Bulletin, No. 242, Dec., 75 (TP 235).
  of lubricating oils
    Wheeling steel method (Metzger). Published in ASTM Bulletin, No. 242, Dec., 77 (TP 237).
Detergents
  determination of
    alkyl aryl sulfonates (Kelley, Blank, Thompson, and Fine). Published in ASTM Bulletin, No. 237, April, 70 (TP 90) (*R*).
    phosphate species
      by ion exchange chromatography (Kolloff). Published in ASTM Bulletin, No. 237, April, 74 (TP 94) (*R*).
  fundamental research (Harris). Published in ASTM Bulletin, No. 238, May, 18.
  Report of Committee D-12, 489.
  synthetic
    methods for analysis. Bibliographical abstracts (*issued as STP 150-C*).
Die Casting
  Report of Committee B-6, 262.
District Activities
  Published in ASTM Bulletin, No. 235, Jan., 22; No. 236, Feb., 20; No. 237, April, 30; No. 238, May, 20; No. 239, July, 37; No. 240, Sept., 18; No. 241, Oct., 20; No. 242, Dec., 40.

District Activities (*continued*)
  Report of Administrative Committee on District Activities, 106.
  Report of Board of Directors, 71.
  Summary of Proceedings, 71.
Dosimetry
  Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 46, 47, 48 (*to be published in STP 276*):
    calorimetry
      reactor monitoring (Hall, Klaver, Carroll, and Bolt)
    concluding remarks (Holden)
    electrical properties of Teflon
      effects of gamma radiation (Loy, Jr.)
    gamma, recent developments (Taimuty)
    in Europe and the USSR (Guill and Moteff)
    in the United States (Tschaeche)
    introduction (Calkins)
    pressure vessel steels
      irradiation (Trudeau)
      neutron radiation embrittlement at 500 and 650 F (Alger and Skupien)
    reactor neutron spectra
      measuring techniques (Trice)
    radiation effects
      dynamic, testing methods (Newell, Kerlin, Bauerlein, and Barrows)
      in metals
        atomistic interpretation (Sosin)
    terminology
      standardization for gamma and electron beam radiation sources (Danald)
    unmanned nuclear propelled vehicles (Stiehl, Jr.)
    uranium burnup
      radiochemical determination (Sunderman)
    X-rays and gamma rays
      environmental measurement by removal dose (Hickmott)

E

Education in Materials
  Symposium. Summary of Proceedings of *STP 263*, 1330:
    engineering education
      ASTM views (LaQue).
      (luncheon address) (Walker).
      in metals
        bearing of challenges of the design engineer (Warren).
      projected degree in materials engineering (Shank) (*not to be published*).
    introduction (Woods).
    materials science
      and education and industry
        solid state physics (Goldman) (*not to be published*).
    nature and properties of materials—Disc. of Report of ASEE Follow-up Subcommittee on Materials (Murphy).
    viewpoint
      of an educator (Alexander).
      of industry (Wood).
      of the civil engineer (Lepper, Jr.).
      (Walker). Published in ASTM Bulletin, No. 239, July, 8.
Efflorescence
  of masonry mortar
    test for (Rogers). Published in ASTM Bulletin, No. 235, Jan., 31 (TP 1). Disc., No. 240, Sept., 45 (TP 175).
Electrical Contact Materials
  Report of Committee B-4, 202.
Electrical Heating Alloys
  Report of Committee B-4, 202.
Electrical Instruments
  activated carbon
    organic contamination adsorption (Laskie). Published in ASTM Bulletin, No. 241, Oct., 41 (TP 205).
Electrical Insulating Liquids and Gases
  Report of Committee D-27, 550.
  story of Subcommittee IV, Committee D-9, 1916–1959 (Johnson). Published in ASTM Bulletin, No. 238, May, 25.
  See also Cables *and* High Voltage.
Electrical Insulating Materials
  asbestos
    iron content rate (Nicodemus). Published in ASTM Bulletin, No. 237, April, 62 (TP 82). Disc., 67 (TP 87).
  coated fabrics
    evaluation of thermal stability by curved electrodes
      dielectric breakdown
        suggested method. Published in 1959 Compilation of ASTM Standards on Electrical Insulating Materials, p. 747.
      dielectric prooftest
        suggested method. Published in 1959 Compilation of ASTM Standards on Electrical Insulating Materials, p. 740.
  coated sleevings
    evaluation of thermal stability
      dielectric breakdown
        suggested method. Published in 1959 Compilation of ASTM Standards on Electrical Insulating Materials, p. 752.
  dust and fog tracking resistance
    suggested method. Published in 1959 Compilation of ASTM Standards on Electrical Insulating Materials, p. 739.
  laminated thermoset
    resistivity (Schlabach). Published in ASTM Bulletin, No. 238, May, 33 (TP 101) (*R*).
  liquids and gases
    new ASTM committee (D-27). Published in ASTM Bulletin, No. 237, April, 48.
    1916–1959, story of ASTM Subcommittee IV, Committee D-9 (Johnson). Published in ASTM Bulletin, No. 238, May 25.
  psychrometric table for relative humidity
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix II), p. 778 (*also in Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 567 (1931)).

  - Report of Committee D-9, 468.
  - Report of Committee D-20, 532.
  - usefulness of tests
    - proposed recommendations for writing statements on. Published in 1959 Compilation of ASTM Standards on Electrical Insulating Materials, p. 737.
  - *See also* Cables, High Voltage, Insulating Materials, *and* Suggested Methods.
- Electrical Insulation
  - Report of Committee F-1, 628.
- Electrical Testing
  - Report of Committee D-9, 468.
- Electrodeposited Coatings
  - Report of Committee B-8, 315.
  - standardizing preparation
    - corrosion (Graham and Pinkerton). Report of Committee B-8 (appendix), 317 (*R*).
  - supplementary coatings
    - Report of Committee B-8, 315.
- Electroless Nickel Plating
  - Symposium. Summary of Proceedings of *STP 265*, 1073:
    - advantages and limitations (Saubestre).
    - applications (Safranek).
    - bibliography of references (Waite).
    - characteristics of deposits (Metzger, Jr.).
    - chemical reactions (Gutzeit).
    - history of the process (Brenner).
    - patent situation (Gutzeit).
    - processing procedures (Krieg).
    - test methods (Cohen).
- Electromagnetic Testing
  - Report of Committee B-1, 163.
  - Report of Committee E-7, 593.
- Electronics Industry
  - Symposium on Materials in the Electronics Industry. Summary of Proceedings of Third Pacific Area Meeting, 43 (*not to be published*):
    - electronic ceramics (Koenig).
    - electron-tube and semiconductor materials (Standing and Biondi) (Committee F-1).
    - insulation and printed wiring (Endicott).
    - magnetic materials and testing (Eberly).
- Electron Metallography
  - Report of Committee E-4, 586.
  - Symposium. Summary of Proceedings of *STP 262*, 1072:
    - carbon replica technique (Pellier).
    - composition of γ′ phase in high-temperature alloys (Birks and Seebold) (*not to be published*).
    - dislocations in thin metal foils (Fisher and Szirmae).
    - effect of heat on a cast nickel alloy (Bieber and Decker).
    - introduction (Nielsen).
    - microscopy of tin plate (Stoll).
    - microstructure of precipitation-hardenable austenitic and nickel-base alloys (Progress Report of Subcommittee XI of ASTM Committee E-4).
    - of neutron-irradiated steels (McCartney and Trozzo).
    - of steels (Pellissier).
    - phases in high-temperature alloys (Mihalisin).
    - routine diffractometry
      - instrumental modifications (Grieger).
      - structure analysis (Grieger).
    - structure and growth of tin oxide (Trozzo).
- Electron Tube Materials
  - Report of Committee F-1, 628.
- Elevated Temperature
  - effect on creep and creep-rupture properties
    - of ferritic and austenitic stainless steels (Garofalo), 973 (*R*). Disc., 983.
  - effect on properties
    - of Cufenloy 30 (Cu-Ni alloy) (Simmons, Sirois, Williams, and Jaffee), 1035, (*R*). Disc., 1046.
  - fatigue tests
    - on cast irons (Felgar), 767.
  - influence
    - of stress concentration
    - on creep of metals of temperature conditions
    - literature surveys (Brown, Jr., Manson, Sachs and Sessler) (*Issued as STP 260*).
  - irradiation at
    - design problems (Stang, Goldthwaite, and Dunnington). Published in ASTM BULLETIN, No. 242, Dec., 54 (TP 214).
  - mechanical properties
    - of high purity lead and 0.058 per cent copper-lead alloy (Tietz), 1052. Disc., 1071.
  - Report of ASTM-ASME Committee on Effect of Temperature on the Properties of Metals, 339.
- Emission Spectroscopy
  - Report of Committee E-2, 569.
- Enamel, Porcelain
  - Report of Committee C-22, 418.
- Engine Antifreezes
  - Report of Committee D-15, 503.
- Explosibility Tester
  - for air-suspended solids (Box). Published in ASTM BULLETIN, No. 241, Oct. 34 (TP 198) (*R*).
- Exposure of Nonferrous Metals
  - Report of Committee B-3, 174.

F

- Fabrics
  - Report of Committee D-13, 492.
  - *See also* Textiles.
- Fatigue
  - cast irons
    - elevated temperatures (Felgar), 767.
  - cold-rolled aluminum sheet
    - fluctuating tension tests (Lipsitt, Forbes, and Baird), 734 (*R*). Disc., 748.
  - current activities, Report of Subcommittee III of ASTM Committee E-9. Published in ASTM BULLETIN, No. 235, Jan., 26.
  - depletion of ductility
    - under stress cycling (Marin, Borachia, and Rimrott), 662 (*R*). Disc., 673.
  - effect of temper-embrittlement
    - on properties of 3140 steel (Mikus and Siebert) (*R*). Disc. (*publication pending*).
  - fracture
    - of AISI 347 stainless steel (Martin and Brinn), 677 (*R*). Disc., 685.

Fatigue (*continued*)
  high-stress, low-cycle properties
    notched alloy specimens (Yukawa and McMullin). Published in ASTM Bulletin, No. 241, Oct., 38 (TP 202).
  high temperature alloys
    static and dynamic stresses (Hawkes and Ek). Published in ASTM Bulletin, No. 235, Jan., 46 (TP 16). Disc., 51 (TP 21).
  laminate sheet structures
    aluminum (Baird, Forbes, and Lipsitt), 755 (*R*).
  manual on testing (*issued as STP 91*)
    proposed revision of section II. Report of Committee E-9 (appendix I), 596.
  metals
    basic research (Dolan). Published in ASTM Bulletin, No. 240, Sept., 24 (*R*).
    stepwise load increase test (Enomoto), 711 (*R*). Disc., 721.
  Report of Committee E-9, 595.
  SAE and vacuum-melted steels
    for aircraft bearings (Sachs, Sell, and Brown, Jr.), 635 (*R*). Disc., 658.
  strain-cycled 2S aluminum
    density measurements (Tavernelli and Coffin, Jr.), 952.
  symbols, list of. Report of Committee E-9 (appendix II), 604.
  Symposium on Fatigue of Aircraft Structures —Summary of Proceedings of Third Pacific Area Meeting, 46, 48, 49 (*to be issued as STP 274*):
    crack propagation
      method for predicting rate of (McEvily, Jr. and Illg).
    cycle ratio in cumulative damage (Grover).
    improvement of strength
      by production methods of cold working joints subjected to fretting (Waters).
    loading spectra
      for testing aircraft components and structures (McCulloch).
    low-cycle, strength of pressurized components (Padlog and Rattinger).
    quantitative and systematic attack on (Haire).
    resistant structures (Smith).
    strength comparisons of forged magnesium alloys (Schuette).
    sonic
      test method (Vreeland).
    testing of
      a helicopter rotor blade (Lischer).
      aluminum alloy specimens (Hardrath and Naumann).
  testing
    at high frequencies (Neppiras), 691 (*R*). Disc., 709.
    servomechanism control system (Danek and Achter). Published in ASTM Bulletin, No. 239, July, 80 (TP 160).
  titanium
    effect of internal heating (Romualdi and D'Appolonia) 723. Disc., 731.
Felt
  fire-retardant properties
    proposed specification. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix VI), p. 802.
FIA Method (with Chromanalyzer)
  for hydrocarbon analysis of olefinic gasolines (Allen and Wood). Published in ASTM Bulletin, No. 238, May, 49 (TP 117).
Fibers
  Report of Committee D-13, 492.
Filterability Test
  for aviation turbine fuels
    proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix VI), p. 1142.
Filter Block, Clay
  Report of Committee C-15, 402.
Films
  supported
    wet adherence to various bases (Diamond). Published in ASTM Bulletin, No. 236, Feb., 48 (TP 58) (*R*).
  See also Wet Abrasion.
Finances
  ASTM, 1958. Published in ASTM Bulletin, No. 236, Feb., 10.
  auditor's report, 81.
    Report of Board of Directors, 76.
Fineness
  of fiber
    in wool fabric and yarns
      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XV), p. 835.
Fireclay Brick
  Report of Committee C-8, 383.
Fire Testing
  endurance test of wall panels
    prediction of temperature rise (Neisel). Published in ASTM Bulletin, No. 239, July, 66 (TP 146).
  for treated felt
    proposed specification. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix VI), p. 802.
  Report of Committee E-5, 588.
Flame Photometry
  Symposium on Spectroscopy (Session III)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 269*):
    analytical—new developments (Gilbert, Jr.)
    determination of tetraethyllead in gasoline (Buell).
    from medical and biological standpoints (Thiers).
Flexure Properties
  plastics
    three-point test (Doyle and Olszewski). Published in ASTM Bulletin, No. 241, Oct., 44 (TP 208).
Floor Coverings
  sandpaper-abrasion resistance (Gavan). Pub-

lished in ASTM BULLETIN, No. 238, May, 44 (TP 112).
Floor Polishes
  Report of Committee D-21, 542.
Floor Wax
  Report of Committee D-21, 542.
Flue Lining
  Report of Committee C-15, 402.
Fluid Bulk Modulus
  proposed definitions. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix IV), p. 1138.
Fluid-Flow Method
  limestone aggregates (Dolch), 1204 (*R*). Disc., 1214.
Fluorescent Indicator Test
  using chromanalyzer and method D 1319 apparatus
    cooperative comparative data. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix V), p. 1140.
Fly Ash
  pulverized coal
    fundamental characteristics (Minnick), 1155.
Formaldehyde Finishes
  on textiles
    identification
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix VII), p. 803.
Friction Meter
  for evaluating properties of hand of fabrics
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix V), p. 798.
Fuel Elements
  irradiated
    use of ultrasonics (Fouts). Published in ASTM BULLETIN, No. 242, Dec., 60 (TP 220).
Fuels
  Report of Committee D-2, 431.
  See also Filterability Test, Gasolines, Luminometer Numbers, *and* Oils.
Fuel Oils
  chromanalyzer and method D 1319 apparatus
    cooperative comparative data. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix V), p. 1140.
  proposed method
    for smoke density in the flue gases from distillate fuels. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix I), p. 1127.
  See also Oils.

G

Gamma Rays
  and X-rays
    environmental measurement by removal dose (Hickmott)—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 276*).
Gaseous Fuels
  Report of Committee D-3, 447.
Gasolines
  olefinic, hydrocarbon analysis
    FIA method with chromanalyzer (Allen and Wood). Published in ASTM BULLETIN, No. 238, May, 49 (TP 117).
  proposed method
    for motor or research octane number of small samples. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix II), p. 1131.
  Report of Committee D-2, 431.
  See also Petroleum *and* Oils.
Glass
  bond characteristics, with resin (McGarry). Published in ASTM BULLETIN, No. 235, Jan., 63 (TP 33).
  See also Window Assemblies.
Glass Fiber
  reinforced laminates
    flexural modulus (Chambers and McGarry). Published in ASTM BULLETIN, No. 238, May, 38 (TP 106).
  reinforced plastics
    failure mechanism (Desai and McGarry). Published in ASTM BULLETIN, No. 239, July, 76 (TP 156).
  See also Glass Textiles *and* Laminates.
Glass Textiles
  basic properties of fibers
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix I), p. 777.
  determining average quality
    specification of number of tests
      proposed recommended practice. Published in 1959 Compilation of ASTM Standards on Textile Materials (appendix IV), p. 791.
Gloss
  Report of Committee E-12, 606.
Grain Size
  Report of Committee E-4, 586.
Graphite
  as a fuel matrix (Riley)—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 276*).
  silicon carbide clad (Taylor and McMurtry)—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 276*).
Grout
  asphaltic (Butterfield and Haney)—Symposium on Bituminous Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 280*).
  field test specimens
    for reinforced grouted brick masonry (Kelch)—Symposium on Masonry Materials—Summary of Proceedings of

Grout (*continued*)
Third Pacific Area Meeting, 39 (*publication pending*).
in reinforced brick masonry
values in diagonal tension and shear (Kelch)—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*publication pending*).
structural bond to concrete block (Copeland)—Symposium on Masonry Materials—Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*).
"Guarded Hot Plate" Method
textiles
thermal insulating properties (Wing and Monego). Published in ASTM BULLETIN, No. 241, Oct., 29 (TP 193).
Gypsum
control in portland cement (Tremper), 1101. Disc., 1107.
crystals
influence in plaster setting (O'Kelley). Published in ASTM BULLETIN, No. 237, April, 55 (TP 75) (*R*). Disc., No. 243, Jan., 1960, 51 (TP 9).
Report of Committee C-11, 393.

H

Hand of Fabrics
evaluating properties
proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix V), p. 798.
Hardness
cemented carbides (Caugherty, Oatman, and Reen), 1261. Disc., 1275.
Report of Committee E-1, 557.
timber
improved and semiautomatic method (Schniewind). Published in ASTM BULLETIN, No. 236, Feb., 57 (TP 67).
Hardware
Report of Committee A-5, 127.
tests
Report of Subcommittee XVI of Committee A-5, 133.
Heat Exchanger Tubes
residual stresses (Nelson). Published in ASTM BULLETIN, No. 240, Sept., 39 (TP 169).
Heavy Liquid Separation Method
proposed tentative test
for determining cement content in freshly mixed concrete. Published in ASTM BULLETIN, No. 239, July, 48.
High Temperature
See Elevated Temperature *and* Temperature Effect.
See also Electron Metallography *and* Plastics.
High-Temperature Alloys
static and dynamic stresses (Hawkes and Ek). Published in ASTM BULLETIN, No. 235, Jan., 46 (TP 16). Disc., 51 (TP 21).
High Voltage
cable insulation
Symposium. Summary of Proceedings of *STP 253*, 1340.
cable oils
utility viewpoint (Gates and Gillette).
why and how of tests (Blodgett).
paper for cables (Delmar).
Highway Construction
research for (Armstrong). Published in ASTM BULLETIN, No. 241, Oct., 14.
Highway Materials
ASTM standards (Hveem)—Symposium on Standards—Summary of Proceedings of Third Pacific Area Meeting, 55 (*publication pending*).
Report of Committee D-4, 449.
Honors and Awards
Report of Board of Directors, 75.
Honorary Members of ASTM
elected 1959. Published in ASTM BULLETIN, No. 239, July, 23.
Humidity
relative
psychrometric table
proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix II), p. 778 (*also in Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 567).
rust protection by metal preservatives
proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix VII), p. 1146.
Hydraulic Fluids
bulk modulus
proposed definitions. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix IV), p. 1138.
(Peeler and Green). Published in ASTM BULLETIN, No. 235, Jan., 51 (TP 21) (*R*).
Symposium. Summary of Proceedings of Third Pacific Area Meeting, 42, 43, 44 (*issued as STP 267*):
developments and evaluation methods (Session II).
aircraft (Peeler and Kovacich).
emulsion type fire resistant (Francis and Sipple).
high performance fluids
for military aircraft and commercial industrial applications (Smith).
spray flammability test (Rowand).
use in high pressure shipboard equipment (King and Coil).
problems and trends (Session I).
are they holding their own? (Sharpe and Henrikson).
automotive central hydraulic systems
fluid requirements (Risk).
is hydraulics out of this world? (Keller).
objectives and activities of Technical Committee N (ASTM Committee D-2) (Millett).
Hydrocarbons
aromatic
Report of Committee D-16, 505.
chromanalyzer and method D 1319 apparatus
cooperative comparative data. Published in

1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix V), p. 1140.
naphthalene
  in jet fuel
    proposed method of test by ultraviolet spectrophotometry. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix III), p. 1135.
particulate matter
  proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix IX), p. 1163.
Hydrogen Bubble Chambers
  low-temperature materials problems (Birmingham, Chelton, Mann, and Hernandez). Published in ASTM BULLETIN, No. 240, Sept., 34 (TP 164) (*R*).
8-Hydroxyquinoline Method
  portland cement
    determination of magnesium oxide (Berman). Published in ASTM BULLETIN, No. 237, April, 51 (TP 71).

I

Ignition Loss
  in portland blast-furnace slag cements (Chaiken). Published in ASTM BULLETIN, No. 238, May, 53 (TP 121).
Incinerators
  municipal, refractories for (Engdahl and Sullivan). Published in ASTM BULLETIN, No. 240, Sept., 52 (TP 182).
Inclusion Content
  Report of Committee E-4, 586.
Industrial Chemicals
  new ASTM Committee (E-15). Published in ASTM BULLETIN, No. 237, April, 48.
Industrial Wastes
  Report of Committee D-19, 523.
Industrial Waste Water
  Manual on Industrial Water and Industrial Waste Water (*issued as separate publication, ASTM STP No. 148-D*).
  procedure for determination of precision of ASTM Committee D-19 methods. To be published in 1960 Compilation of ASTM Standards on Industrial Water (*see also* Manual on Industrial Water and Industrial Waste Water, *STP 148-D*, p. 203).
  Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 50, 51 (*to be published as STP 273*):
    conservation and pollution control programs cooling towers (Finnerty).
    determination of radioactive materials (Barker).
    industrial waste
      effects on waters for irrigation use (Wilcox).
      radioactive, disposal of at Hanford (Schwendiman, Brown, Honstead, Linderoth, and Pearce).
    municipal supply of Coalinga, Calif.
      batch-type and continuous electric membrane demineralizers (Katz).
    municipal waste treatment (Kinney).
    oil field brines
      disposal in Central Valley, Calif. (Morris, Jr.).
    saline
      solar distillation
        present status and material problems (Lappala, Yaeger, and Bjorksten).
    sea
      field testing of corrosion inhibitors (Wright).
    treatment for once-through reactor cooling (Richman).
Infrared Absorption
  Symposium on Spectroscopy (Session V)—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 269*):
    chemical (Potts).
    preparing non-brittle solid organic samples (Strait and Hrenoff).
    quantitative (Wilson and Steele).
    ten years of aid by Committee E-13 (Robey).
Infrared Balance
  moisture test
    proposed method. 1959 Compilation of ASTM Standards on Soaps and Other Detergents (*issued as separate reprint*).
Infrared Spectroscopic Methods
  proposed recommended practice (*issued as separate reprint*).
Ink
  viscosity measurement by use of viscometer
    modified McMichael
      for consistency of printing inks and ink vehicles (Cummings)—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).
    Stormer
      for testing rotogravure and flexographic inks (Cummings)—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).
Insulating Materials
  electrical
    asbestos
      iron content rate (Nicodemus). Published in ASTM BULLETIN, No. 237, April, 62 (TP 82). Disc., 67 (TP 87).
    Report of Committee D-9, 468.
  Symposium on Electrical Insulating Materials—Summary of Proceedings of Third Pacific Area Meeting, 41 (*publication pending*):
    Committee D-9
      activities of (Endicott).
    improved oils (Furby and Hanly).
    International Electrotechnical Commission Dealing with Electrical Insulating Materials
      activities of (Scott).

Insulating Materials (*continued*)
    magnet wire insulations and epoxy encapsulating resins
        compatibility of (Lee).
  thermal
      Report of Committee C-16, 406.
  See also Electrical Insulating Liquids, Electrical Insulating Materials *and* High Voltage.
Insulating Wire and Cable
  Report of Committee D-11, 479
Interferometry
  infrared
    thickness measurement of organic coatings (Brightwell). Published in ASTM BULLETIN, No. 237, April, 67 (TP 87).
Interlaboratory Tests
  evaluation of methods (Mandel and Lashof). Published in ASTM BULLETIN, No. 239, July, 53 (TP 133).
International Relations
  Report of Administrative Committee on Standards, 99.
International Standards
  ACR notes (Townsend). Published in ASTM BULLETIN, No. 236, Feb., 15.
In-Place Strength Test
  concrete (Benjamin and Ratliff, Jr.). Published in ASTM BULLETIN, No. 241, Oct., 23 (TP 187). Disc., 25 (TP 189).
Ion Exchange Chromatography
  analysis of commercial sodium tripolyphosphate (Kolloff). Published in ASTM BULLETIN, No. 237, April, 74 (TP 94) (*R*).
  determination of phosphate species in detergents (Kolloff). Published in ASTM BULLETIN, No. 237, April, 74 (TP 94) (*R*).
Iron
  in asbestos materials for electrical insulation (Nicodemus). Published in ASTM BULLETIN, No. 237, April, 62 (TP 82). Disc., 67 (TP 87).
  See also Cast Iron.

J

Jet Fuels
  chromanalyzer and method D 1319 apparatus
    cooperative comparative data. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix V), p. 1140.
  filterability test
    proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix VI), p. 1142.
  luminometer numbers
    proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix VIII), p. 1157.
  particulate matter in hydrocarbons
    proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix IX), p. 1163.
  proposed method
    for naphthalene hydrocarbons by ultraviolet spectrophotometry. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix III), p. 1135.

K

Kerosine
  filterability test
    proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix VI), p. 1142.
  luminometer numbers
    proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix VIII), p. 1157.
  naphthalene hydrocarbons in jet fuel
    proposed method of test by ultraviolet spectrophotometry. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix III), p. 1135.
  proposed method
    for smoke density in the flue gases from distillate fuels. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix I), p. 1127.
Knit Fabrics
  dimensional changes in laundering
    proposed method. Published in 1959 Compilation of ASTM Standards on Textile Materials (appendix X), p. 809.
Knock Characteristics, Tests for
  proposed method
    for motor or research octane number of small samples. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix II), p. 1131.

L

Laboratory Apparatus
  standardization of measurement units (Ruh, Moran, and Thompson). Published in ASTM BULLETIN, No. 240, Sept., 31 (TP 161).
Laminates
  fiberglass reinforced plastics
    failure mechanisms (Desai and McGarry). Published in ASTM BULLETIN, No. 239, July, 76 (TP 156).
    flexural modulus (Chambers and McGarry). Published in ASTM BULLETIN, No. 238, May, 38 (TP 106).
  flexure properties
    three-point test (Doyle and Olszewski). Published in ASTM BULLETIN, No. 241, Oct., 44 (TP 208).
  resin-glass
    bond characteristics (McGarry). Published in ASTM BULLETIN, No. 235, Jan., 63 (TP 33).
  thermosetting
    temperature dependence of electrical resistivity (Schlabach). Published in ASTM

Bulletin, No. 238, May, 33 (TP 101) (*R*).

- *See also* Plastics.

Latex
- water permeability studies (Simon)—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).

Lead
- high purity and 0.058 per cent copper alloy
  - mechanical properties at elevated temperatures (Tietz), 1052. Disc., 1071.
- Report of Committee B-2, 171.

Leather
- Report of Joint Committee on Leather, 555.

Lectures
- Gillett
  - dislocations in plastic deformation (Fisher). Published in ASTM Bulletin, No. 237, April, 7; No. 239, July, 6 (synopsis); No. 241, Oct., 6 (*published as separate pamphlet*).
- Marburg
  - new polymers—new problems (Mark). Published in ASTM Bulletin, No. 237, April, 7; No. 239, July, 6 (synopsis); No. 241, Oct., 6 (*published as separate pamphlet*); to be published in ASTM Bulletin, No. 245, April, 1960.

Light Metals
- Report of Committee B-7, 263.

Lime
- for stabilization
  - powder *versus* slurry (Davidson, Noguera, and Sheeler). Summary of Proceedings of *STP 254*, 1335.
- hydrated
  - calcium oxide in (Moorehead and Taylor). Published in ASTM Bulletin, No. 236, Feb., 45 (TP 55).
- Report of Committee C-7, 381.

Limestone
- fluid-flow measurements (Dolch), 1204 (*R*). Disc., 1214.

Lithium
- purification
  - solubility of nitrogen and oxygen (Hoffman)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*published in STP 272*).
- Report of Committee B-2, 171.

Long-Range Planning
- Report of Long-Range Planning Committee, 63.

Low-Temperature
- hydrogen bubble chambers
  - materials problem (Birmingham, Chelton, Mann, and Hernandez). Published in ASTM Bulletin, No. 240, Sept., 34 (TP 164) (*R*).
- tensile properties of copper and bronze (McClintock, Van Gundy, and Kropschot). Published in ASTM Bulletin, No. 240, Sept., 47 (TP 177).
- *See also* Temperature Effect.

Lubricants
- demulsibility
  - of high viscosity oils (Brennan). Published in ASTM Bulletin, No. 242, Dec., 75 (TP 235).
  - of lubricating oils
    - Wheeling steel method (Metzger). Published in ASTM Bulletin, No. 242, Dec., 77 (TP 237).
- Report of Committee D-2, 431.
- winter grade motor oils
  - pour stability characteristics
    - proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix X), p. 1169.
- *See also* Oils.

Lumber
- *See* Wood.

Luminometer Numbers
- of aviation turbine fuels
  - proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants, (appendix VIII), p. 1157.

M

Magnesium
- alloys
  - atmospheric exposure tests
    - ASTM test sites (Adams and Dougherty). Report of Committee B-7 (appendix III), 289.
  - spectrochemical analysis
    - suggested method
      - by the point-to-plane spark technique using a grating spectrograph (Murphy). Report of Committee E-2 (appendix II), 577.
- forged alloys
  - fatigue-strength comparisons (Schuette)—Symposium on Fatigue of Aircraft Structures—Summary of Proceedings of Third Pacific Area Meeting, 49 (*to be published in STP 274*).
- in galvanic cells
  - formed by marine exposure coupled (Gallaccio) (*issued as STP 255*).
- oxide
  - determination in portland cement
    - by 8-hydroxyquinoline method (Berman). Published in ASTM Bulletin, No. 237, April, 51 (TP 71).
- Report of Committee B-6, 262.
- Report of Committee B-7, 263.

Magnetic Particle
- Report of Committee E-7, 593.

Magnetic Properties
- copper alloys (Parker, Winter, and LaPorta). Report of Committee B-5 (appendix III), 230 (*R*).
- Report of Committee A-6, 156.

Magnetic Resonance
- Symposium on Spectroscopy (Session IV)—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 269*):
  - conjugated organic compounds

Magnetic Resonance (*continued*)
      band nomenclature for the ultraviolet spectra (Forbes and Shilton).
    Maser applications and traveling-wave techniques (Siegman)
    nuclear (Shoolery).
    polynuclear heteroaromatic compounds
      electron spin resonance spectra of free radicals (Tuck and Schieser).
    the unpaired electron via EPR (Sands).
Magnetic Testing
  Report of Committee A-6, 156.
Management and Responsibilities
  changes (Nason). Published in ASTM BULLETIN, No. 242, Dec., 28.
  purchasing (Cresap, Jr.). Published in ASTM BULLETIN, No. 242, Dec., 35.
Marine Atmosphere Exposure
  coupled to form galvanic cells involving magnesium (Gallaccio) (*issued as STP 255*).
Masonry
  Report of Committee C-12, 395.
  See also Building Constructions.
Material Questions. Published in ASTM BULLETIN, No. 238, May, 12; No. 239, July, 35; No. 240, Sept., 13; No. 241, Oct., 13; No. 242, Dec., 24.
Materials
  education in (Walker). Published in ASTM BULLETIN, No. 239, July, 8; No. 240, Sept., 20.
  in aerospace age (Schriever). Published in ASTM BULLETIN, No. 242, Dec., 16.
  needed in 1970. AIA report. Published in ASTM BULLETIN, No. 240, Sept., 28.
  sciences
    new ASTM division. Published in ASTM BULLETIN, No. 236, Feb., 5; Report of Board of Directors, 61.
  See also Education in Materials.
Maturity Test
  for cotton fibers
    causticaire method
      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XIV), p. 830.
Meetings
  Annual Meeting
    1959. Summary of Proceedings, 1.
      Report of Board of Directors, 69.
      Published in ASTM BULLETIN, No. 235, Jan., 20; No. 236, Feb., 6; No. 237, April, 5; No. 238, May, 5; No. 239, July 5; No. 240, Sept., 5.
  Committee Week
    1959. Published in ASTM BULLETIN, No. 235, Jan., 19; No. 237, April, 33; No. 242, Dec., 32.
  National Conference on Standards. Published in ASTM BULLETIN, No. 235, Jan. 20.
  Third Pacific Area National Meeting
    Summary of Proceedings, 36.
    Published in ASTM BULLETIN, No. 236, Feb., 11; No. 238, May 8; No. 239, July, 32; No. 240, Sept., 21; No. 242, Dec., 5.
Membership
  Report of Board of Directors, 73.
Mercury
  in porosimetry, size measurement (Winslow and Shapiro). Published in ASTM BULLETIN, No. 236, Feb., 39 (TP 49) (*R*).
Metal Cleaners
  Report of Committee D-12, 489.
Metal Coatings
  Report of Committee A-5, 127.
  Report of Committee C-22, 418.
  See also Coatings.
Metallography
  Report of Committee E-4, 586.
  See also Electron Metallography.
Metal Powders
  Report of Committee B-9, 330.
Metals
  newer
    Symposium. Summary of Proceedings of Third Pacific area Meeting, 50, 51, 53, 54 (*issued as STP 272*):
      nuclear and light metals (Session II).
        beryllium
          elevated temperature properties
            effect of purity and manufacturing variables (Beaver, O'Rourke, and Hurd).
          QMV
            stress-rupture and creep properties (Hurd, O'Rourke, and Beaver).
        yttrium and alloys
          mechanical properties (Carlson, Bare and Gibson.)
        zircaloy-2
          tensile and corrosion properties
            effects of heat treatments (Goodwin, Rubenstein, and Shubert).
        zirconium and alloy
          in the Naval reactor program (Kall).
      processing of (Session III).
        chromium
          deoxidation and denitrification
            effect on low temperature properties (Levingston and Grant).
        columbium and tantulum
          fabrication (McCullough).
        lithium
          purification
            solubility of nitrogen and oxygen (Hoffman).
        tungsten
          and properties (Toensing).
        zircaloy
          analysis of
            by coulometric determination of tin with electrically generated iodine (Freund and Caton).
      refractory metals, properties (Session I).
        columbium (niobium).
          workability and mechanical properties
            effect of oxygen and nitrogen (Begley and France).
        molybdenum and molybdenum-base alloy sheet
          mechanical properties (Semchyshen and Barr).

    platinum-group
      tensile properties (Holden, Douglass and Jaffee).
    tantalum
      high-temperature mechanical properties (Holden, Jaffee, and Schwartzberg).
    vacuum and controlled atmosphere
      universal high-temperature testing machine (Manjoine, France, and Begley).
  See also Alloys, Aluminum, Beryllium, Bronze, Copper, Iron, Metallography, Nickel, Steel, Titanium, Zinc, etc.
Metals Analysis
  by radioisotopes
    Symposium on Radioisotopes in Metals Analysis and Testing. Summary of Proceedings of *STP 261*, 1339:
      applications
        neutron activation
          of traces of molybdenum in tungsten (Cosgrove).
        of aluminum
          experiences with activation (Lewis).
        the (n, alpha) reaction (Busshey).
        training industrial personnel (Danforth).
      by analysis of
        instrumentation and techniques (Emmons)
      by radioactivation (Morrison).
      dilution methods
        refereeing (Rosenblum).
      introduction (Beeghley).
      nucleonics (Meinke).
Mica
  Report of Committee D-9, 468.
Microscopy
  Report of Committee E-1, 557.
  Symposium. Summary of Proceedings of *STP 257*, 1074:
    chemical
      in the optical industry (Crossmon).
    electron
      application to the petroleum industry (Allred).
      in the study of mineral and ceramics (Comer).
    evaluation of cotton finishing treatments (Rollins, deGruy, Tripp and Moore).
    introduction (Morehead).
    measurement
      with phase and interference microscopes (Richards).
    performance characteristics
      of objectives, eyepieces and illuminators (Zieler).
    study of
      failure in polymers (Newman).
      opaque minerals in reflected light (Cameron).
    synthetic fibers, structure of (Scott).
Missile and Aircraft Materials
  stainless-steel honeycombs (Gilbraith). Published in ASTM BULLETIN, No. 240, Sept., 51 (TP 181).
Missile Industry
  ablation and erosion in the rocket and the missile environment (Herzog and Donaldson)—Symposium on Thermal Ablation—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 279*).
  Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 39, 41 (*to be published as STP 278*):
    mobile field testing
      of missiles and aircraft (Barath and Hagemaier).
    radiography
      high-energy
        in the 6- to 30-Mev range (Bly and Burrill).
      of large solid propellant rocket motors (Criscuolo, Polansky, Halloway, and Dyer).
      of weldments in motion (Hitt and Hagemaier).
    ultrasonic method
      gaging of missile components during manufacture (Bloch).
      inspection of adhesion bonded aluminum sandwich structures (Kammerer).
      standards for evaluation of missile materials and components (Adams).
      to detect, count and measure particle size of contamination in fluids (Albertson).
Moisture
  brick
    rate of absorption (Hodgins). Published in ASTM BULLETIN, No. 241, Oct., 27 (TP 191).
  surface
    measurement instrumentation (Sereda). Published in ASTM BULLETIN, No. 238, May, 61 (TP 129).
  See also Moisture Test.
Moisture Regain
  wool and its products
    recommendation for standard. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix IX), p. 808.
Moisture Test
  infrared balance
    proposed method. 1959 Compilation of ASTM Standards on Soaps and Other Detergents (*issued as separate reprint*).
Molybdenum
  and molybdenum-base alloy sheet
    mechanical properties (Semchyshen and Barr)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting (*published in STP 272*).
  Report of Committee B-2, 171.
Mortars
  control of SO_3 in portland cement
    short-time tests (report of Working Committee on SO_3 Content). Report of Committee C-1 (appendix I), 349.

Mortars (*continued*)
efflorescence (Rogers). Published in ASTM BULLETIN, No. 235, Jan., 31 (TP 1). Disc., No. 240, Sept., 45 (TP 175).
field test specimens
for reinforced grouted brick masonry (Kelch)—Symposium on Masonry Materials—Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*).
plasticity and workability (Conrow), 1075. Disc., 1086.
portland cement
release of alkalines (Hansen). Published in ASTM BULLETIN, No. 236, Feb., 35 (TP 45).
Report of Committee C-3, 378.
Report of Committee C-12, 395.
structural bond to concrete block (Copeland)—Symposium on Masonry Materials—Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*).
testing for efflorescence (Rogers)—Symposium on Masonry Materials—Summary of Proceedings of Third Pacific Area Meeting, 39 (*publication pending*).

N

National Officers of ASTM
elected 1959. Published in ASTM BULLETIN, No. 239, July, 20.
Naval Stores
Report of Committee D-17, 515.
New Members. Published in ASTM BULLETIN, No. 235, Jan., 80; No. 236, Feb., 72; No. 237, April, 88; No. 238, May, 70; No. 239, July, 88; No. 240, Sept., 70; No. 241, Oct., 64; No. 242, Dec., 94.
Neutron Irradiation
Report of Committee A-1, 110.
Nickel
Report of Committee B-2, 171.
See also Electroless Nickel Plating *and* Electron Metallography.
Nickel Alloys
Report of Committee B-2, 171.
See also Electron Metallography.
Nitric-Hydrofluoric Test
intergranular corrosion resistance
type 316 L stainless steel (Warren). Discussion. Published in ASTM BULLETIN, No. 235, Jan., 44 (TP 14).
Notch Effect
bending and tension
determination of energy release (Lubahn), 885 (*R*). Disc., 914.
creep-rupture behavior
austenitic stainless steel (Garofalo), 957 (*R*).
Notched Geometry
effect of depth in bending and tension
determination of energy release rate (Lubahn), 885 (*R*). Disc., 914.
effect on tensile properties at low temperatures of annealed titanium (Geil and Carwile), 985 (*R*). Disc., 1013.
See also Notch Effect.

Nuclear Applications
ceramics
Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 39, 40 (*to be published in STP 276*).
beryllium oxide (Hove).
forming of particles
for fuels, control and burnable poison dispersion components (Ploetz and Muccigrosso).
fuel elements
dry pressing to close tolerances (Hartwig)
graphite matrix (Riley).
silicon carbide clad (Taylor and McMurtry).
U_3O_8-Al
fabrication by extrusion (Noland, Walker and Hymes).
urania-alumina and boron carbide burnable poisons
fabrication and evaluation (Wisnyi and Taylor).
refractory uranium compounds—preparation and properties (Snyder).
Report of Committee B-7, 263.
Nuclear Energy
fuel elements
aluminum alloys
long-range segregation of uranium (Peckner). Published in ASTM BULLETIN, No. 242, Dec., 51 (TP 211).
irradiation at high temperature
design problems (Stang, Goldthwaite, and Dunnington). Published in ASTM BULLETIN, No. 242, Dec., 54 (TP 214).
uranium slug and aluminum can
testing of unbonded areas by ultrasonics (Fouts). Published in ASTM BULLETIN, No. 242, Dec., 60 (TP 220).
Nuclear Radiation
Report of Committee B-5, 205.

O

Octane Number Test
motor or research
proposed method for small samples. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix II), p. 1131.
Oils
demulsibility
high viscosity (Brennan). Published in ASTM BULLETIN, No. 242, Dec., 75 (TP 235).
for high voltage cables
utility viewpoint (Gates and Gillette). Summary of Proceedings of *STP 253*, 1340.
why and how of tests (Blodgett). Summary of Proceedings of *STP 253*, 1340.
insulating, improved (Furby and Hanly)—Symposium on Electrical Insulating Materials—Summary of Proceedings of Third Pacific Area Meeting, 41 (*publication pending*).

lubricating
  Wheeling steel method (Metzger). Published in ASTM BULLETIN, No. 242, Dec., 77 (TP 237).
  See also Fiterability Test, Kerosine, Lubricants, *and* Pour Point.
Optical Emission
  Symposium on Spectroscopy (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 269*):
    atomic spectroscopy (McNally, Jr.).
    radioactive materials (Conway).
    spectrochemical analysis (Fry).
Organic Coatings
  thickness measurement
    by infrared interferometry (Brightwell). Published in ASTM BULLETIN, No. 237, April, 67 (TP 87).

P

Paint
  discussion of work of Committee D-1 (Gloger)—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).
  latex and latex paint film
    water permeability studies (Simon)—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).
  metal protective
    exposure fence testing (Lominska)—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).
  pigment colors
    lightfastness testing (Spengeman and Wormald)—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).
  Report of Committee D-1, 420.
Paper
  AMS 3722
    for honeycomb core in sandwich structure (Zemer)—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 42 (*published in STP 270*).
  for high voltage cables (Delmar). Summary of Proceedings of *STP 253*, 1340.
  psychrometric table for relative humidity
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix II), p. 778 (*also in Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part 1, p. 567).
  Report of Committee D-6, 457.
Paperboard
  psychrometric table for relative humidity
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix II), p. 778 (*also in Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part 1, p. 567).
  Report of Committee D-6, 457.
Papers and Publications
  Report of Administrative Committee on Papers and Publications, 102.
Particle Size
  of contamination in fluids
    ultrasonic method to detect, count and measure (Albertson)—Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 278*).
Particulate Matter
  in hydrocarbons
    proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix IX), p. 1163.
Petroleum
  foreign (Lenzen)—Petroleum and Chemical Industry Luncheon Address—Summary of Proceedings of Third Pacific Area Meeting, 37 (*not to be published*).
  See Gasoline, Kerosine, Oils *and* Proposed Methods.
Petroleum Products
  Report of Committee D-2, 431.
  See also Proposed Method.
Photometric Methods of Chemical Analysis
  proposed method
    for naphthalene hydrocarbons in jet fuel. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix III), p. 1135.
Pigments
  lightfastness testing (Spengeman and Wormald)—Session on Paint and Related Products—Summary of Proceedings of Third Pacific Area Meeting, 38 (*publication pending*).
  Report of Committee D-1, 420.
Pipe
  clay, Report of Committee C-4, 380.
  concrete, Report of Committee C-13, 396.
  plastic, Report of Committee D-20, 532.
Pitch
  coal tar flat (Fair, Jr. and Chamberlain)—Symposium on Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 280*).
  industrial (Pittman and Donegan)—Symposium on Waterproofing and Roofing Materials—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 280*).
  roofing materials
    Report of Committee D-8, 466.
Planoflex Apparatus
  for evaluating properties of hand of fabrics
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix V), p. 798.
Plaster
  physical changes in setting
    influence of gypsum crystals (O'Kelly). Published in ASTM BULLETIN, No. 237,

Plaster (*continued*)
        April, 55 (TP 75) (*R*). Disc., No. 243, Jan., 1960, 51 (TP 9).
Plastics
    capsules
        for high-temperature irradiations (Stang, Goldthwaite, and Dunnington). Published in ASTM BULLETIN, No. 242, Dec., 54 (TP 214).
    fiberglass, reinforced laminates
        failure mechanisms (Desai and McGarry). Published in ASTM BULLETIN, No. 239, July, 76 (TP 156).
        flexural modulus (Chambers and McGarry). Published in ASTM BULLETIN, No. 238, May, 38 (TP 106).
    flexure properties
        three-point test (Doyle and Olszewski). Published in ASTM BULLETIN, No. 241, Oct., 44 (TP 208).
    polyethylene
        control of thermal history of test specimens (Birks and Rudin). Published in ASTM BULLETIN, No. 242, Dec., 63 (TP 223).
    polymers
        organic-inorganic systems
            for elevated temperature resistant metal-to-metal adhesives (Janis)—Symposium on Adhesion and Adhesives—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 271*).
        post-irradiation effects
            Symposium. Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 276*):
                and monomers (Degering, Caldarella, and Mancini)
                free radicals in irradiated materials
                    electron spin resonance studies (Wall).
                in irradiated materials
                    significance to ASTM (Ballantine).
                polysterene and poly(methyl methacrylate) (Parkinson and Binder).
                silicones
                    dielectric properties (Currin).
    psychrometric table for relative humidity
        proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix II), p. 778 (*also in Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 567).
    reinforced
        Symposium. Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 279*):
            evaluation
                for high temperature structural applications (Hribar and Dowell).
                of reinforcements, problems (Silver, Erickson, and Perry).
            filament wound materials
                test for mechanical properties (McCarthy).
            permeability of materials
                under high pressures and at various temperatures (Epstein and Gary, Jr.).
            testing and specifying—introductory remarks (Perry).
        testing under simulated re-entry conditions
            determination of steady-state heat of ablation (John and Schick) (Part I);
            plasma jet calibration (John and Bade) (Part II)—Symposium on Thermal Ablation—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 279*).
    Report of Committee D-9, 468.
    Report of Committee D-20, 532.
    sandwich construction
        glass-fabric plastic honeycomb core (Marshall)—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 270*).
        polysterene foam core panels in roofs and walls (Edberg)—Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings of Third Pacific Area Meeting, 44 (*to be published in STP 270*).
    testing oven, modified
        for accelerated aging of vinyl plastic compounds (Royen). Published in ASTM BULLETIN, No. 243, Jan., 1960, 43 (TP 1).
    Vicat softening point
        modified testing apparatus (Ehlers). Published in ASTM BULLETIN, No. 236, Feb., 54 (TP 64). Disc., No. 241, Oct., 40 (TP 204).
    See also Laminates, Polymers, *and* Resins
Platinum
    metal group
        tensile properties (Holden, Douglass, and Jaffee)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 272*).
Polarization Resistance
    relation to corrosion rate (Stern and Weisert), 1280 (*R*). Disc., 1291.
Polyethylene
    test specimens
        control method (Birks and Rudin). Published in ASTM BULLETIN, No. 242, Dec., 63 (TP 223).
    See also Plastics.
Polymers
    new—new problems (Mark) (Marburg Lecture). Published in ASTM BULLETIN, No. 239, July, 6 (synopsis); No. 241, Oct., 6 (synopsis); to be published in ASTM BULLETIN, No. 245, April, 1960 (*also published as separate pamphlet*).
    organic-inorganic systems
        for elevated temperature resistant metal-to-metal adhesives (Janis)—Symposium on Adhesion and Adhesives—Summary of Proceedings of Third Pacific Area Meeting, 52 (*to be published in STP 271*).
    post-irradiation effects

Symposium. Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 276*):
    and monomers (Degering, Caldarella, and Mancini).
    free radicals in irradiated materials
        electron spin resonance studies (Wall).
    in irradiated materials
        significance to ASTM (Ballantine).
    polysterene and poly(methyl methacrylate) (Parkinson and Binder).
    silicones
        dielectric properties (Currin).
  See also Plastics.
Porcelain Enamel
  Report of Committee C-22, 418.
Porosimetry
  measurement of pore-size
    by mercury penetration (Winslow and Shapiro). Published in ASTM BULLETIN, No. 236, Feb., 39 (TP 49) (*R*).
Post-Irradiation
  Symposium on Post-Irradiation Effects in Polymers—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 276*):
    dielectric properties
      of silicones (Currin).
    effects
      in irradiated materials
        significance to ASTM (Ballantine).
      monomers and polymers (Degering, Caldarella and Mancini).
    free radicals in irradiated materials
      electron spin resonance studies (Wall).
    oxidation and molecular weight changes
      in polystyrene and poly (methyl methacrylate) (Parkinson and Binder).
Pour Point
  winter grade motor oils
    proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix X), p. 1169.
Powdered Materials
  explosibility tester (Box). Published in ASTM BULLETIN, No. 241, Oct., 34 (TP 198) (*R*).
President's Address
  science—impact on ASTM (Woods), 56; published in ASTM BULLETIN, No. 239, July, 9.
Preservatives (Wood)
  Report of Committee D-7, 460.
Product Appearance
  standardization and communicating
    by visual aids
      Symposium on New Visual Aids for Standardizing and Communicating Product Appearance. Summary of Proceedings of *STP 258*, 1331:
        ASTM methods and standards (Nimeroff).
        inspection by closed circuit television (Vendeland).
        in the textile industry (Goldberg).
        introduction (Hemphill).
        specification and control by color photography (Kane).
        summation (Ingle).
Production
  versus tests (Chartz)—Electronics Industry Luncheon Address—Summary of Proceedings of Third Pacific Area Meeting, 37 (*not to be published*)
Proposed Definitions
  of fluid bulk modulus. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix IV), p. 1138.
Proposed Method
  cement content of fresh concrete. Published in ASTM BULLETIN, No. 239, July, 48.
  infrared spectrophotometry
    for *ortho*-xylene, *meta*-xylene, *para*-xylene and ethylbenzene. Report of Committee D-16 (appendix), 510.
  petroleum products
    filterability of aviation turbine fuels. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants, p. 1142.
    luminometer numbers of aviation turbine fuels. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants, p. 1157.
    motor or research octane number of small samples. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants, p. 1131.
    naphthalene hydrocarbons in jet fuel by ultraviolet spectrophotometry. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants, p. 1135.
    particulate matter in hydrocarbons. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants, p. 1163.
    pour stability characteristics of winter grade motor oils. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants, p. 1169.
    rust protection by metal preservatives in the humidity cabinet. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants, p. 1146.
    smoke density in the flue gases from distillate fuels. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants, p. 1127.
  soaps
    rapid determination of solids by infrared moisture balance. Published in 1959 Compilation of ASTM Standards on Soap and Other Detergents (*issued as separate reprint*)
  textile materials
    cotton content of asbestos textile materials (referee method). Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 812.
    dimensional changes in knitted fabrics in

Proposed Method (*continued*)
    laundering and dimensional restorability of the laundred fabric. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 809.
  estimating maturity and weight per unit length of cotton fibers by the causticaire method. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 830.
  evaluation of properties related to the hand of soft-finished woven fabrics. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 798.
  fire-retardant properties of treated felt. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 802.
  identification of finishes on textiles. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 803.
  length and length distribution of cotton fibers by the short method array. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 824.
  psychrometric table for relative humidity. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 778 (*see also* ASTM *Proceedings*, Vol. 31, Part I, p. 567).
  recovery of textile fabrics from creasing using the roller pressure apparatus. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 806.
  sampling and preparation of test specimens from wool fabric and yarns for determination of fiber fineness. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 835.
  strength of cotton fibers using a finite gage length (flat bundle method). Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 817.
  yarn number conversion table. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 784.
 water, industrial
  procedure for determination of precision of Committee D-19 methods. To be published in 1960 Compilation of ASTM Standards on Industrial Water (*see also* Manual on Industrial Water and Industrial Waste Water, *ASTM STP 148-D*, p. 203).
Proposed Recommendations
 affecting standards
  concrete pipe. Report of Committee C-13 (appendix), 398.
  light metals and alloys, cast and wrought. Report of Committee B-7 (appendix I), 267.
  steel. Report of Committee A-1 (appendix), 116.
  textile materials. Report of Committee D-13 (appendix), 494.
 for the establishment of standard moisture content for wool and its products. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 808.
Proposed Recommended Practices
 quantitative analysis
  infrared
   general techniques. Report of Committee E-13 (appendix I), 610 (*R*).
  ultraviolet
   general techniques. Report of Committee E-13 (appendix II), 622 (*R*).
 spectroscopy
  general techniques of infrared quantitative analysis (*issued as separate ASTM reprint*).
  general techniques of ultraviolet quantitative analysis (*issued as separate ASTM reprint*).
 textile materials
  for calculating number of tests to be specified in determining average quality. Published in 1959 Compilation of ASTM Standards on Textile Materials, p. 791.
Proposed Specification
 sealing compounds (polysulfides). Published in ASTM BULLETIN, No. 242, Dec., 46.
Protein Materials
 Report of Committee D-25, 548.
Publications
 new ASTM publications. Published in ASTM BULLETIN, No. 235, Jan., 16; No. 236, Feb., 12; No. 237, April, 26; No. 238, May, 14; No. 239, July, 36; No. 240, Sept., 16; No. 242, Dec., 26.
 ASTM publications in 1959–1960. Published in ASTM BULLETIN, No. 241, Oct., 5.

Q

Quality Control
 Report of Committee E-11, 605.
Quick Lime
 Report of Committee C-7, 381.

R

Radiation
 test methods
  Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42, 43, 44 (*to be issued as STP 268*):
   grain boundary segregation studies
    by activation (Schultz).
   instrumental methods of analysis (Manov and Beaufait).
   magnesium oxide in portland cement
    determination by radiometric method (Howes, Jr., Brown, Sunderman, Pobereskin, and Schall).
   organic sheet materials
    permeability (Pobereskin).
   radioactive ring wear testing
    in railroad Diesel locomotives (Jursch, Pinotti, and Jones).
   soil
    detergency test
     by calcium-45-tagged clay (Hensley and Inks).
    moisture and density measurement
     by nuclear method (Kuranz).

        radiotagged fatty
            removal from glass surface (Harris and Anderson).
        uniformity of coated fabrics (Foster).
        uniformity of mixing
            rapid determination by tracer methods (Main).
        very thin vacuum evaporated metal films
            tracer techniques (Preuss).
Radiation Effect
    Report of Committee A-10, 158.
    Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 46, 47, 48 (*to be published in STP 276*):
        calorimetry
            reactor monitoring (Hall, Klaver, Carroll, and Bolt).
        concluding remarks (Holden).
        dosimetry
            gamma, recent developments (Taimuty).
            in Europe and the USSR (Guill and Moteff).
            in the United States (Tschaeche).
        dynamic, testing methods (Newell, Kerlin, Bauerlein, and Barrows).
        electrical properties of Teflon
            effects of gamma radiation (Loy, Jr.).
        in metals
            atomistic interpretation (Sosin).
        introduction (Calkins).
        pressure vessel steels
            irradiation (Trudeau).
            neutron radiation embrittlement at 500 and 650 F (Alger and Skupien).
        reactor neutron spectra
            measuring techniques (Trice).
        terminology
            standardization for gamma and electron beam radiation sources (Danald).
        unmanned nuclear propelled vehicles (Stiehl, Jr.).
        uranium burnup
            radiochemical determination (Sunderman).
        X-rays and gamma rays
            environmental measurement by removal dose (Hickmott).
Radiography
    high-energy
        in the 6- to 30-Mev range (Bly and Burrill) —Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 278*).
    of large solid propellent rocket motors (Criscuolo, Polansky, Halloway, and Dyer)—Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 278*).
    of weldments in motion (Hitt and Hagemaier) —Symposium on Nondestructive Testing in the Missile Industry—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 278*).
    Report of Committee E-7, 593.
Radioisotopes
    test methods
        Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42, 43, 44 (*to be published as STP 268*):
            grain boundary segregation studies
                by activation (Schultz).
            instrumental methods of analysis (Manov and Beaufait).
            magnesium oxide in portland cement
                determination by radiometric method (Howes, Jr., Brown, Sunderman, Pobereskin, and Schall).
            organic sheet materials
                permeability (Pobereskin).
            radioactive ring wear testing
                in railroad Diesel locomotives (Jursch, Pinotti, and Jones).
            soil
                detergency test
                    by calcium-45-tagged clay (Hensley and Inks).
                moisture and density measurement
                    by nuclear method (Kuranz).
                radiotagged fatty
                    removal from glass surface (Harris and Anderson).
            uniformity of coated fabrics (Foster).
            uniformity of mixing
                rapid determination by tracer methods (Main).
            very thin vacuum evaporated metal films
                tracer techniques (Preuss).
    Symposium on Radioisotopes in Metals Analysis and Testing, Summary of Proceedings of *STP 261*, 1339.
        analysis of
            instrumentation and techniques (Emmons).
        applications in analysis
            neutron activation
                of traces of molybdenum in tungsten (Cosgrove).
            of aluminum
                experiences with activation (Lewis).
            the (n, alpha) reaction (Busshey).
            training industrial personnel (Danforth).
        dilution methods
            refereeing (Rosenblum).
        introduction (Beeghley).
        metals analysis by radioactivation (Morrison).
        nucleonics in analysis (Meinke).
Railroads
    research, report. Published in ASTM BULLETIN, No. 237, April, 31.
Random Samples
    current materials news. Published in ASTM BULLETIN, No. 235, Jan., 30; No. 236, Feb., 61; No. 237, April, 81; No. 240, Sept., 61; No. 241, Oct., 47; No. 242, Dec., 83.
Residual Stresses
    in heat-exchanger tubes (Nelson). Published in ASTM BULLETIN, No. 240, Sept., 39 (TP 169).

- Reference Blocks
  - for ultrasonic inspection (Panian and Van Valkenburg), 1237 (*R*). Disc., 1259.
- Reference Method
  - portland cement mortar
    - expansion in water and contraction in air. Report of Committee C-1 (appendix I), 366.
- Refractories
  - for municipal incinerators
    - fluctuating temperature conditions (Engdahl and Sullivan). Published in ASTM BULLETIN, No. 240, Sept., 52 (TP 182).
  - Report of Committee C-8, 383.
- Research
  - ASTM reports file. Published in ASTM BULLETIN, No. 241, Oct., 12.
  - cooperation on problems
    - ACR notes (Shobert). Published in ASTM BULLETIN, No. 239, July, 50.
  - cryogenic
    - for hydrogen bubble chambers (Birmingham, Chelton, Mann, and Hernandez). Published in ASTM BULLETIN, No. 240, Sept., 34 (TP 164) (*R*).
  - fatigue of metals (Dolan). Published in ASTM BULLETIN, No. 240, Sept., 24 (*R*).
  - fundamental and committee work (Harris). Published in ASTM BULLETIN, No. 238, May, 18.
  - highway construction (Armstrong). Published in ASTM BULLETIN, No. 241, Oct., 14.
  - in materials
    - proposed changes in standards (Marin)—Symposium on Standards—Summary of Proceedings of Third Pacific Area Meeting, 55 (*publication pending*).
  - materials needed in 1970. Report of AIA. Published in ASTM BULLETIN, No. 240, Sept., 28.
  - OTS reports. Published in ASTM BULLETIN, No. 235, Jan., 92; No. 236, Feb., 78; No. 237, April, 95; No. 238, May, 75; No. 239, July, 111; No. 240, Sept., 76, No. 241, Oct., 62; No. 242, Dec., 100.
  - railroads. Published in ASTM BULLETIN, No. 237, April, 31.
  - Report of Administrative Committee on Research, 107.
  - reports file. Published in ASTM BULLETIN, No. 241, Oct., 12.
  - role in ASTM, ACR notes (Alden). Published in ASTM BULLETIN, No. 235, Jan., 25.
  - *See* ACR Notes.
- Resin
  - epoxy encapsulating
    - compatibility with magnet wire insulations (Lee)—Symposium on Electrical Insulating Materials—Summary of Proceedings of Third Pacific Area Meeting, 41 (*publication pending*).
  - glass
    - bond characteristics, with (McGarry). Published in ASTM BULLETIN, No. 235, Jan., 63 (TP 33).
  - tetrafluoroethylene
    - determination of crystallinity (McCrum). Published in ASTM BULLETIN, No. 242, Dec., 80 (TP 240).
  - *See also* Plastics.
- Resin-Glass
  - bond characteristics (McGarry). Published in ASTM BULLETIN, No. 235, Jan., 63 (TP 33).
- Road and Paving Materials
  - Symposium. Summary of Proceedings of Third Pacific Area Meeting, 48, 50 (*to be published as STP 277*):
    - asphalt
      - chemical composition
        - influence on performance (Rostler and White).
      - mixtures
        - temperature effect on flexibility (Monismith).
    - mineral aggregates
      - degradation (Minor).
        - test for production of plastic fines (Ekse and Morris).
    - Symposium on the Zaca-Wigmore Experimental Test Road
      - cooperative test series on asphalts (Skog).
      - correlation of durability tests with field hardening (Simpson, Miles, and Griffin).
      - progress report (Hveem, Zube, and Skog).
- Road Materials
  - Report of Committee D-4, 449.
  - Report of Committee D-18, 518.
  - *See also* Bituminous Paving Materials *and* Highway Materials.
- Rockwell Hardness Testing
  - brass strip
    - effect of gage (Trout). Report of Committee B-5 (appendix II), 212.
  - *See also* Hardn[illegible]Testing.
- Roofing Materia[illegible]
  - Report of Committee D-8, 466.
- Rosin
  - Report of Committee D-17, 515.
- Rotor Forgings
  - large Ni-Mo-V
    - effect of composition and processing variables (Kramer and Steiner), 916.
- Rubber
  - curing times
    - Mooney test (Juve). Published in ASTM BULLETIN, No. 239, July, 71 (TP 151).
  - Report of Committee D-11, 479.
- Rusting
  - in the humidity cabinet
    - protection by metal preservatives
      - proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix VII), p. 1146.

S

- Sandwich Construction
  - structural
    - durability and weathering
      - Symposium on Durability and Weathering of Structural Sandwich Constructions—Summary of Proceedings

of Third Pacific Area Meeting, 42, 44 (*to be published as STP 270*):
adhesive bonded structures (Tuckerman).
aircraft applications (Reese).
AMS 3722 paper honeycomb core (Zemer).
applications in building construction (Biggs).
glass-fabric plastic honeycomb core (Marshall).
polysterene foam core panels in roofs and walls (Edberg).
temperature rise due to solar heating (Rider).
Report of Committee C-19, 412.
Science Fairs
ACR notes (McPherson). Published in ASTM BULLETIN, No. 237, April, 29.
Sealants
for concrete joints
testing machine (Schutz and Van Hauter). Published in ASTM BULLETIN, No. 241, Oct., 42 (TP 206).
proposed specification
for building trade
polysulfides. Published in ASTM BULLETIN, No. 242, Dec., 46.
Semiconductors
Report of Committee F-1, 628.
Shipping Containers
Report of Committee D-10, 477.
Shrinkage Test
knit fabrics
proposed method. Published in 1959 Compilation of ASTM Standards on Textile Materials (appendix X), p. 809.
Siding Materials
Report of Committee D-8, 466.
Silicon Steel
Report of Committee A-6, 156.
Silicones
for highway light reflectance (Cahn and Mackey, Jr.). Published in ASTM BULLETIN, No. 235, Jan., 37 (TP 7). Disc., 41 (TP 11).
post-irradiation dielectric properties (Currin)—Symposium on Post-Irradiation Effects in Polymers—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 276*).
Silk
basic properties of fibers
proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix I), p. 777.
Slag
blast-furnace, portland cement
ignition loss (Chaiken). Published in ASTM BULLETIN, No. 238, May, 53 (TP 121).
Smoke Density Test
for the flue gases from distillate fuels
prosposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix I), p. 1127.
SO_3 Content
in portland cement. Report of Committee C-1 (appendix I), 349.
Soaps
Report of Committee D-12, 489.
See also Proposed Method.
Soaps and Detergents
infrared moisture balance
proposed method. 1959 Compilation of ASTM Standards on Soaps and Other Detergents (*issued as separate reprint*).
Sodium Hydroxide Swelling Test
for cotton fibers
causticaire method
proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XIV), p. 830.
Softening Point
plastics
modified Vicat type apparatus (Ehlers). Published in ASTM BULLETIN, No. 236, Feb., 54 (TP 64). Disc., No. 241, Oct., 40 (TP 204).
Soils
detergency test
by calcium-45-tagged clay (Hensley and Inks)—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 268*).
moisture and density measurement
by nuclear method (Kuranz)—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 268*).
radiotagged fatty
removal from glass surface (Harris and Anderson)—Symposium on Applied Radiation and Radioisotope Test Methods—Summary of Proceedings of Third Pacific Area Meeting, 42 (*to be published in STP 268*).
Report of Committee D-18, 518.
Symposium on Soils for Engineering Purposes—Summary of Proceedings of Third Pacific Area Meeting, 52, 54—Summary of Proceedings of *STP 254*, 1335:
classification triangle
based on Unified Soil Classification System (USCS) (McMinn).
dynamic characteristics
determination of *in situ* (Bernhard) (*presented by title only*)
evaluation and design of concrete pavements
repetitive plate load tests (Gordon).
excess hydrostatic pressures
measurement (Weber, Jr.).
moisture contents
effect of temperature determined by centrifuge and tension techniques (Prill and Johnson).
moisture-density relations
laboratory studies (Dawson).
moisture and strength variation

- Soils (*continued*)
    - in a thick uniform clay (Weber, Jr. and Kleiman).
  - particle shape and texture
    - effect on strength of noncohesive aggregates (Morris).
  - triaxial compression tests
    - using variable lateral pressure (Meese and Long).
  - Symposium on Time Rates of Loading in Soil Testing. Symposium on Atterberg Limits. Session on Soils. Summary of Proceedings of *STP 254* (Soils—1959), 1335:
    - Atterberg limits
      - correlation with geology of deep cores (Johnson and Morris).
      - history and development of tests (Bauer).
      - introduction (Sowers).
      - liquid limit tests
        - investigation of (Dawson).
        - one-point
          - mechanical method (Joslin and Davis).
          - procedure (Eden).
        - penetration tests (Sowers, Vesic, and Grandolfi).
        - proposed universal standards (Morris, Ulp, and Spinna).
        - use of grooving tools (Mitchell).
      - plastic limit
        - cube and standard thread methods (Abdun-Nur).
    - cement
      - granular mixtures for base-courses (Abrams).
    - lime
      - for stabilization
        - powder *versus* slurry (Davidson, Noguera, and Sheeler).
    - sensitive clay
      - preconsolidation pressure
        - improved determination (Hamilton and Crawford).
    - time rates of loading in testing
      - apparatus for repeated load tests (Seed and Fead).
      - cohesive soil
        - dynamic and static resistance (Housel).
      - consolidation
        - automation in testing (Karol).
        - time effects on clays (Leonards and Ramiah).
      - introduction (Burmister)
      - sensitive clay
        - influence of strain-rate on effective stresses (Crawford).
      - strain-rate behavior
        - clay and organic solids (Burmister).
      - triaxial shear tests
        - adjusting strain-rates for pore pressure measurements (Ellis and Holtz).
      - visco-elastic stress-strain laws (Schiffman).
- Solder
  - Report of Committee B-2, 171.
- Solids Content Test
  - by infrared moisture balance
    - proposed method. 1959 Compilation of ASTM Standards on Soaps and Detergents (*issued as separate reprint*).
- Solvents
  - halogenated organic
    - Report of Committee D-27, 549
  - Report of Committee D-1, 420.
- Soviet Russia
  - a new industrial threat (Ewell). Published in ASTM BULLETIN, No. 239, July, 43.
- Spectrochemical Analysis
  - index to the literature (Scribner and Meggers) (*issued as STP 41-D*).
- Spectrography
  - *See* Spectroscopy.
- Spectrophotometry
  - methods of analysis for organic ions
    - bibliography (Mellon and Bly) (*issued as STP 125-A*).
- Spectroscopic Excitation
  - Symposium. Summary of Proceedings of *STP 259*, 1338:
    - introductory remarks (Scribner).
    - effects of gaseous atmospheres (Andermann and Kemp).
    - properties of new or modified sources (Margoshes).
    - the matrix effect (Hurwitz).
- Spectroscopy
  - Report of Committee E-2, 569.
  - Report of Committee E-13, 607.
  - Symposium. Summary of Proceedings of Third Pacific Area Meeting, 40, 41, 43, 44, 49, 51 (*issued as ASTM STP 269*):
    - general topics (Session VI).
      - analysis of complex hydrocarbon systems (Coggeshall and Hubis).
      - molecular spectroscopy in the USSR (Rea).
      - three European meetings in 1959 (Brattain).
      - ultraviolet spectroscopy
        - aromaticity; absorption errors (Friedel).
    - infrared absorption (Session V).
      - chemical (Potts).
      - preparing non-brittle solid organic samples (Strait and Hrenoff).
      - quantitative (Wilson and Steele).
      - ten years of aid by Committee E-13 (Robey).
    - magnetic resonance (Session IV).
      - conjugated organic compounds
        - band nomenclature for the ultraviolet spectra (Forbes and Shilton).
      - Maser applications and traveling-wave techniques (Siegman).
      - nuclear (Shoolery).
      - polynuclear heteroaromatic compounds
        - electron spin resonance spectra of free radicals (Tuck and Schieser).
      - the unpaired electron via EPR (Sands).
    - optical emission (Session I).
      - atomic spectroscopy (McNally, Jr.).
      - radioactive materials (Conway).
      - spectrochemical analysis (Fry).
    - ultraviolet absorption and flame photometry (Session III).

    analytical—new developments (Gilbert, Jr.).
    determination of tetraethyllead in gasoline (Buell).
    from medical and biological standpoints (Thiers).
    in the region of 175–200 mμ (Kaye).
    (Vandenbelt).
  X-ray (Session II).
    application to unsolved problems in geochemistry (Adler).
    fluorescence
      basic practices (Birks).
      instrumentation (Kemp).
    spectrography
      quantitative light element analysis
        Fe to Mg for portland cement (Curley).
 See also Emission Spectroscopy, Flame Photometry, Infrared Absorption, Magnetic Resonance, Proposed Method, Proposed Recommended Practices, *and* Suggested Methods.
Standardization
  actions on standards. No 236, Feb., 14; No. 238, May, 15; No. 239, July 30; No. 240, Sept., 15; No. 241, Oct., 22; No. 242, Dec. 44.
  building materials (Legget). Published in ASTM BULLETIN, No. 237, April, 36.
  federal government standards changes. Published in ASTM BULLETIN, No. 235, Jan., 94; No. 236, Feb., 93; No. 237, April, 110; No. 238, May, 93; No. 239, July, 108; No. 240, Sept., 100; No. 241, Oct., 78; No. 242, Dec., 119.
  in India (Pabst, Jr.). Published in ASTM BULLETIN, No. 242, Dec., 31.
  instrument and laboratory apparatus (Ruh, Moran, and Thompson). Published in ASTM BULLETIN, No. 240, Sept., 31 (TP 161).
  international (Townsend). Published in ASTM BULLETIN, No. 236, Feb., 15.
  new nickel oxide standards. Published in ASTM BULLETIN, No. 238, May, 17.
  new tentative and standards. Published in ASTM BULLETIN, No. 239, July, 30.
  of preparation of electrodeposits for corrosion testing (Graham and Pinkerton). Report of Committee B-8 (appendix), 317 (*R*).
  of terminology for gamma and electron beam radiation sources (Danald)—Symposium on Radiation Effect and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 276*).
  product appearance
    by visual aids
      Symposium on New Visual Aids for Standardizing and Communicating Product Appearance. Summary of Proceedings of *STP 258*, 1331:
        ASTM methods and standards (Nimeroff).
        inspection by closed circuit television (Vendeland).
        in the textile industry (Goldberg).
        introduction (Hemphill).
        specification and control by color photography (Kane) (*not to be published*).
        summation (Ingle).
  Report of Administrative Committee on Standards, 95.
  Symposium on Standards—Are Changes in Order? Summary of Proceedings of Third Pacific Area Meeting, 55 (*publication pending*):
    ASTM, highway materials (Hveem)
    based upon research in materials
      proposed changes (Marin)
    role in purchasing function (Brunberg)
    sound standards stimulate manufacturing (McCoy)
  See also Proposed Method, Proposed Recommendations, Proposed Recommended Practices, Proposed Specification, Reference Method, *and* Suggested Methods.
Statistical Analysis
  presentation of data
    for determining average quality of textiles
      proposed recommended practice. Published in 1959 Compilation of ASTM Standards on Textile Materials (appendix IV), p. 791.
  procedure for determination of precision of ASTM Committee D-19 methods. To be published in 1960 Compilation of ASTM Standards on Industrial Water (*see also* Manual on Industrial Water and Industrial Waste Water, *STP 148-D*, p. 203).
  Report of Committee E-11, 605.
Statistical Evaluation
  interlaboratory cement tests (Crandall and Blaine), 1129.
Statistics
  application of (Speight). Published in ASTM BULLETIN, No. 236, Feb., 29 (TP 39).
  from certainty to statistics (Squires)—Luncheon Address—Summary of Proceedings of Third Pacific Area Meeting, 37. Published in ASTM BULLETIN, No. 243, Jan., 1960, p. 27.
  theory of extreme values (Mandel). Published in ASTM BULLETIN, No. 236, Feb., 29 (TP 39) (*R*).
  use of
    in interlaboratory tests (Mandel and Lashof). Published in ASTM BULLETIN, No. 239, July, 53 (TP 133).
    in materials specifications (Sheppard and Ginsburg). Published in ASTM BULLETIN, No. 236, Feb., 31 (TP 41).
Steel
  aircraft bearings
    tension, compression, and fatigue (Sachs, Sell and Brown, Jr.), 635 (*R*). Disc., 658.
  AISI 301 and 304L
    effect of cold rolling and stress relief
      notch and smooth tensile characteristics (Espey, Repko, and Brown, Jr.), 816 (*R*). Disc., 833.
  austenitic chromium-nickel stainless
    stress-corrosion cracking (Report) (*issued as STP 264*) (*See* Corrosion).
  cast and forged Cr-Mn-Ni-N

Steel (*continued*)
  property relationships (Haefner, Lahr, Meinhart, and Kanter), 804 (*R*). Disc., 815.
 corrosiveness of test sites
  Report of Subgroup of Subcommittee VII of Committee B-3 (appendix II), 183.
 creep and creep-rupture properties
  effect of environment (Garofalo), 973 (*R*). Disc., 983.
 for pressure vessel
  irradiation (Trudeau)—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 276*).
  neutron radiation embrittlement at 500 and 650 F (Alger and Skupien)—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 276*).
 high strength sheet alloy
  sharp edge notch tensile strength (Espey, Jones, and Brown, Jr.), 837 (*R*). Disc., 872.
 light gage steel building
  lateral shear tests (Holstain)—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 275*).
 Ni-Mo-V forgings
  effect of composition and processing variables (Kramer and Steiner), 916.
 notched alloy specimen
  high-stress, low-cycle fatigue test (Yukawa and McMullin). Published in ASTM BULLETIN, No. 241, Oct., 38 (TP 202).
 paint coatings (Copson and Larrabee). Published in ASTM BULLETIN, 242, Dec., 68 (TP 228) (*R*).
 Report of Committee A-1, 110.
 Report of Committee A-5, 127.
 Report of Committee A-6, 156.
 Report of Committee A-10, 158.
 SAE 4340
  static mechanical properties
   effect of precycling (Marin, Borachia, and Rimrott), 662 (*R*). Disc., 673.
 stainless
  AISI 347
   fatigue fracture (Martin and Brinn), 677 (*R*). Disc., 685.
  AISI type 304L
   with improved strength (Brady), 774. Disc., 785.
  austenitic
   Cr-Mn-C-N
    composition and properties (Kasak, Hsiao, and Dulis), 786. Disc., 802.
   304, 316, 321
    creep-rupture behavior
     notched and unnotched specimens (Garofalo), 957 (*R*).
  corrosion resistance
   nitric acid all-glass multiple-test apparatus (Tisinai and Samans). Published in ASTM BULLETIN, No. 238, May 64 (TP 132).
   honeycombs (Gilbraith). Published in ASTM BULLETIN, No. 240, Sept., 51 (TP 181).
   nitric-hydrofluoric acid evaluation (Warren). Published in ASTM BULLETIN, Disc., No. 235, Jan., 44 (TP 14).
  tensile properties
   at high rates of strain (Austin and Steidel, Jr.), 1292 (*R*). Disc., 1307.
 3140
  fatigue properties
   effect of temper-embrittlement (Mikus and Siebert), (*R*). Disc. (*publication pending*).
 See also Electron Metallography.
Strain Gages
 checking device for indicators (Mills). Published in ASTM BULLETIN, Disc., No. 236, Feb., 47 (TP 57).
 electrical resistance
  false permanent strains (Newton). Published in ASTM BULLETIN, No. 235, Jan., 42 (TP 12). Disc., No. 239, July, 79 (TP 159).
Strain-Rate
 adjustment in triaxial loading
  for pore pressure measurements (Ellis and Holtz). Summary of Proceedings of *STP 254*, 1335.
 behavior of clay and organic solids (Burmister). Summary of Proceedings of *STP 254*, 1335.
 constant
  metals (Hockett), 1309. Disc., 1317.
 in sensitive clay
  influence on effective stresses (Crawford). Summary of Proceedings of *STP 254*, 1335.
 visco-elastic laws (Schiffman). Summary of Proceedings of *STP 254*, 1335.
Stress Concentrations
 influence on creep of metals
  literature surveys (Brown, Jr., Manson, Sachs, and Sessler) (*issued as STP 260*).
Stress-Corrosion Cracking
 austenitic chromium-nickel stainless steels (Report) (*issued as STP 264*) (*See* Corrosion).
Stress Cycling
 effect on static mechanical properties
  of SAE 4340 steel (Marin, Borachia, and Rimrott), 662 (*R*). Disc., 673.
Stress-Strain
 properties
  of selected titanium metals (Klier and Gazzara), 1015 (*R*). Disc., 1029.
Student Employment
 promising trend in, ACR notes (Smith). Published in ASTM BULLETIN, No. 241, Oct., 21.
Suggested Methods
 electrical insulating materials
  dust and fog tracking resistance. Published in 1959 Compilation of ASTM Standards on Electrical Insulating Materials, p. 739.

coated fabrics
evaluation of thermal stability by curved electrodes
dielectric breakdown. Published in 1959 Compilation of ASTM Standards on Electrical Insulating Materials, p. 747.
dielectric prooftest. Published in 1959 Compilation of ASTM Standards on Electrical Insulating Materials, p. 740.
coated sleevings
evaluation of thermal stability
dielectric breakdown. Published in 1959 Compilation of ASTM Standards on Electrical Insulating Materials, p. 752.
spectrochemical analysis
by the point-to-plane spark technique
of antimony (Dunn, Cestaro, and Von Weisenstein). Report of Committee E-2 (appendix I), 573 (*issued as separate reprint*).
magnesium-base alloys
using a grating spectrograph (Murphy). Report of Committee E-2 (appendix II), 577.
zinc-base die casting alloys
and a-c arc technique (*issued as separate reprint*).
Super-Strength Alloys
Report of Committee A-10, 158.
Sulfonates
alkyl aryl
determination by ultraviolet absorption (Kelley, Blank, Thompson, and Fine). Published in ASTM BULLETIN, No. 237, April, 70 (TP 90) (*R*).
Systems of Units
in instrument and laboratory apparatus (Ruh, Moran, and Thompson). Published in ASTM BULLETIN, No. 240, Sept., 31 (TP 161).

T

Tantulum
fabrication (McCullough)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 272*).
high-temperature mechanical properties (Holden, Jaffee, and Schwartzberg)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 51 (*to be published in STP 272*).
Report of Committee B-2, 171.
Tar
Report of Committee D-4, 449.
Report of Committee D-8, 466.
Technical Committee Notes. Published in ASTM BULLETIN, No. 235, Jan., 23; No. 236, Feb., 22; No. 237, April, 40; No. 238, May, 28; No. 239, July, 40; No. 240, Sept., 5; No. 241, Oct., 20; No. 242, Dec., 36.
"Teflon"
TEF-fluorocarbon resin determination of properties by crystallinity (McCrum). Published in ASTM BULLETIN, No. 242, Dec., 80 (TP 240).
Temperature Effect
(literature surveys)
and nonsteady load
on creep of metals
elevated
influence of stress concentrations (Brown, Jr., Manson, Sachs, and Sessler). (*Issued as STP 260*).
on anelastic creep strain (Lubahn). Published in ASTM BULLETIN, No. 239, July, 61 (TP 141). Disc., No. 243, Jan., 1960, 54 (TP 12).
on flexibility characteristics
of asphaltic paving mixtures (Monismith)—Symposium on Road and Paving Materials—Summary of Proceedings of Third Pacific Area Meeting, 50 (*to be published in STP 277*).
on physical and mechanical properties
of Cufenloy 30 (Cu-Ni alloy) (Simmons, Sirois, Williams, and Jaffee), 1035 (*R*). Disc., 1046.
on properties of metals
Report of Joint ASTM-ASME Committee on Effect of Temperature on Properties of Metals, 339.
Report of Committee B-4, 202.
See also Elevated Temperature, High Temperature *and* Low Temperature.
Tension
AISI 301 and 304L steel sheet
effect of cold rolling and stress relief (Espey, Repko, and Brown, Jr.), 816 (*R*). Disc., 833.
engineering materials
at high rates of strain (Austin and Steidel, Jr.), 1292 (*R*). Disc., 1307.
high-speed tester (Strella). Published in ASTM BULLETIN, No. 236, Feb., 59 (TP 69).
high strength steel sheet alloys
sharp edge notch strength (Espey, Jones, and Brown, Jr.), 837 (*R*). Disc., 872.
laminate sheet structures (Baird, Forbes, and Lipsitt), 755 (*R*).
metals
effect of specimen taper on elongation (Kula and Larson). Published in ASTM BULLETIN, No. 238, May, 58 (TP 126). Disc., No. 241, Oct., 37 (TP 201).
notch
determination of energy release (Lubahn), 885 (*R*). Disc., 914.
of textiles, determination of quality
specification of number of tests
proposed recommended practice. Published in 1959 Compilation of ASTM Standards on Textile Materials (appendix IV), p. 791.
properties of steels
for aircraft bearings (Sachs, Sell, and Brown, Jr.) 635 (*R*). Disc., 658.
sharp edge notch
high strength steel sheet alloy (Espey,

Tension (*continued*)
    Jones, and Brown, Jr.), 837 (*R*). Disc., 872.
Termite Control
  in California residential construction (Ebeling)—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 275*).
Testing Apparatus
  plastics
    Vicat softening point (Ehlers). Published in ASTM BULLETIN, No. 236, Feb., 54 (TP 64). Disc., No. 241, Oct., 40 (TP 204).
Testing Machines
  abrader
    for testing floor coverings (Gavan). Published in ASTM BULLETIN, No. 238, May, 44 (TP 112).
  concrete joint sealants (Schutz and Van Hauter). Published in ASTM BULLETIN, No. 241. Oct., 42 (TP 206).
  high-rate tension (Strella), No. 236, Feb., 59 (TP 69).
  physical parameters of metal
    history (1837). Published in ASTM BULLETIN, No. 236, Feb., 26.
  pore-size measurement (Winslow and Shapiro). Published in ASTM BULLETIN, No. 236, Feb., 39 (TP 49) (*R*).
  static and dynamic stresses
    in high-temperature alloys (Hawkes and Ek). Published in ASTM BULLETIN, No. 235, Jan., 46 (TP 16). Disc., 51 (TP 21).
Test Sites
  ASTM. Published in ASTM BULLETIN, No. 236, Feb., 24.
  corrosiveness of
    measured by specimens of steel and zinc. Report of Subgroup of Subcommittee VII of Committee B-3 (appendix II), 183.
Textiles
  asbestos
    cotton content
      referee method
        proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XI), p. 812.
  basic properties of fibers
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix I), p. 177.
  crease test
    by roller pressure apparatus
      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix VII), p. 806.
  determining average quality
    specification of number of tests
      proposed recommended practice. Published in 1959 Compilation of ASTM Standards on Textile Materials (appendix IV), p. 791.
  evaluating properties of hand
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix V), p. 798.
  finishes, identification of
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix VII), p. 803.
  microstrains (Hamburger). Published in ASTM BULLETIN, No. 238, May 23.
  psychrometric table for relative humidity
    proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix II), p. 778 (*also in* ASTM *Proceedings*, Vol. 31, Part I, p. 567).
  thermal insulation
    two methods (Wing and Monego). Published in ASTM BULLETIN, No. 241, Oct., 29 (TP 193).
  wool fabric and yarns
    fiber fineness
      proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XV), p. 835.
  yarn number conversion table. Published in 1959 Compilation of ASTM Standards on Textile Materials (appendix III), p. 784.
  See also Fibers, Proposed Method, Proposed Recommended Practices, *and* Textile Materials.
Textile Materials
  wool and its products
    moisture content
      recommendation for standard. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix IX), p. 808.
  Report of Committee D-13, 492.
Thermal Ablation
  Symposium. Summary of Proceedings of Third Pacific Area Meeting, 49, 51 (*to be published in STP 279*:
    tests and simulation (Session II).
      in the rocket and the missile environment (Herzog and Donaldson).
      reinforced plastics under simulated reentry conditions
        determination of steady-state heat (John and Schick).
        plasma jet calibration (John and Bade).
      under various ablative conditions (King).
    theory and measurements (Session I).
      ablative environments—introductory remarks (Perry).
      principles and capabilities (Steurer).
      surface temperature
        photographic technique (Siviter, Jr.).
      test validity
        related to arc jet kinetics (Buhler).
Thermal Conductivity
  Report of Committee C-16, 406.
Thermal Insulating Materials
  Report of Committee C-16, 406.
  textiles (Wing and Monego). Published in ASTM BULLETIN, No. 241, Oct., 29 (TP 193).

Thermocouples
  high-frequency response system (Tramposch, Papirno, and Girard). Published in ASTM BULLETIN, No. 238, May, 41 (TP 109).
  Report of Committee E-1, 557.
Thermometers
  pour point of lubricants
    proposed method. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix X), p. 1169.
  Report of Committee E-1, 557.
Thermosetting Laminates
  electrical resistivity
    temperature dependence (Schlabach). Published in ASTM BULLETIN, No. 238, May, 33 (TP 101) (*R*).
Thermostat Metals
  Report of Committee B-4, 202.
Thickness Measurement
  organic coatings
    by infrared interferometry (Brightwell). Published in ASTM BULLETIN, No. 237, April, 67 (TP 87).
Thorium
  Report of Committee B-2, 171.
Three-Point Flexure Test
  plastics (Doyle and Olszewski). Published in ASTM BULLETIN, No. 241, Oct., 44 (TP 208).
Tile
  clay, Report of Committee C-15, 402.
  drain, Report of Committee C-15, 402.
Timber
  hardness test
    improved and semiautomatic method (Schniewind). Published in ASTM BULLETIN, No. 236, Feb., 57 (TP 67).
  Report of Committee D-7, 460.
  See also Wood.
Time Rates of Loading
  Symposium. Summary of Proceedings of *STP 254,* 1335:
    apparatus for repeated load tests (Seed and Fead)
    cohesive soil
      dynamic and static resistance (Housel).
    consolidation
      automation in testing (Karol).
      time effects on clays (Leonards and Ramiah).
    sensitive clay
      influence of strain-rate on effective stresses (Crawford).
    strain-rate behavior
      clay and organic solids (Burmister).
    triaxial shear tests
      adjusting strain-rates for pore pressure measurements (Ellis and Holtz).
    visco-elastic stress-strain laws (Schiffman).
Tin
  Report of Committee B-2, 171.
  See also Electron Metallography.
Titanium
  annealed
    tensile properties
      effect of notched geometry at low temperatures (Geil and Carwile), 985 (*R*). Disc., 1013.
  fatigue properties
    effect of internal heating (Romualdi and D'Appolonia) 723. Disc., 731.
  Report of Committee B-2, 171.
  selected metals
    stress-strain properties (Klier and Gazzara), 1015 (*R*). Disc., 1029.
  tensile properties
    at high rates of strain (Austin and Steidel, Jr.), 1292 (*R*). Disc., 1307.
Torsion Modulus
  pendulum for measurement of crystallinity and void content in TFE resins (McCrum). Published in ASTM BULLETIN, No. 242, Dec., 80 (TP 240).
Transformer Oil
  Report of Committee D-27, 550.
Triaxial Loading
  adjusting strain-rates for pore pressure measurements (Ellis and Holtz). Summary of Proceedings of *STP 254,* 1335.
Tube
  See Electron Tube Materials, Heat Exchanger Tubes, *and* Residual Stresses.
Tungsten
  carbides
    Rockwell hardness test (Caugherty, Oatman, and Reen), 1261. Disc., 1275.
  processing and properties (Toensing)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 272*).
  Report of Committee B-2, 171.

U

Ultrasonic Testers
  for high frequency fatigue testing (Neppiras), 691 (*R*). Disc., 709.
Ultrasonics
  inspection
    reference blocks (Panian and Van Valkenburg), 1237 (*R*). Disc., 1259.
  in testing irradiated fuel elements (Fouts). Published in ASTM BULLETIN, No. 242. Dec., 60 (TP 220).
  Report of Committee A-1, 110.
  Report of Committee E-7, 593.
Ultraviolet Absorption
  for determination of alkyl aryl sulfonates (Kelley, Blank, Thompson, and Fine). Published in ASTM BULLETIN, No. 237, April, 70 (TP 90) (*R*).
  spectroscopy
    in the region of 175–200 m (Kaye)—Symposium on Spectroscopy (Session III)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 269*).
    problems in (Vandenbelt)—Symposium on Spectroscopy (Session III)—Summary of Proceedings of Third Pacific Area Meeting, 43 (*to be published in STP 269*).

Ultraviolet Spectroscopic Methods
proposed method
for naphthalene hydrocarbons in jet fuel. Published in 1959 Compilation of ASTM Standards on Petroleum Products and Lubricants (appendix III), p. 1135.
proposed recommended practice (*issued as separate ASTM reprint*).
Units of Measurement
problems in instrument and laboratory apparatus (Ruh, Moran, and Thompson). Published in ASTM BULLETIN, No. 240, Sept., 31 (TP 161).
Uranium
burnup
radiochemical determination (Sunderman)—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 46 (*to be published in STP 276*).
fuel elements
U_3O_8-Al
fabrication by extrusion (Noland, Walker, and Hymes)—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 276*).
urania-alumina and boron carbide burnable poisons
fabrication and evaluation (Wisnyi and Taylor)—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 276*).
long-range segregation in aluminum alloys (Peckner). Published in ASTM BULLETIN, No. 242, Dec., 51 (TP 211).
refractory compounds
preparation and properties (Snyder)—Symposium on Ceramics in Nuclear Energy—Summary of Proceedings of Third Pacific Area Meeting, 39 (*to be published in STP 276*).
Report of Committee B-2, 171.
slug and aluminum can in fuel elements
testing of unbonded areas by ultrasonics (Fouts). Published in ASTM BULLETIN, No. 242, Dec., 60 (TP 220).

V

Vapor Transmission
Report of Committee C-16, 406.
Varnish
insulating
Report of Committee D-9, 468.
Report of Committee D-1, 420.
Vicat Test
softening point of plastics
modified apparatus (Ehlers). Published in ASTM BULLETIN, No. 236, Feb., 54 (TP 64). Disc., No. 241, Oct., 40 (TP 204).
Visual Aids
for standardizing and communicating product appearance
Symposium. Summary of Proceedings of *STP 258*, 1331:
ASTM methods and standards (Nimeroff).
inspection by closed circuit television (Vendeland).
in the textile industry (Goldberg).
introduction (Hemphill).
specification and control by color photography (Kane) (*not to be published*).
summation (Ingle).

W

Wall Panels
fire endurance test
prediction of temperature rise (Neisel). Published in ASTM BULLETIN, No. 239, July, 66 (TP 146).
Water
a new look at the substance (Clarke)—Water Industry Luncheon Address—Summary of Proceedings of Third Pacific Area Meeting, 38. Published in ASTM BULLETIN, No. 243, Jan., 1960, p. 17.
deposits
Symposium on Identification of Water-Formed Deposits. Summary of Proceedings of *STP 256*, 1336:
analysis
and phase identification viewed by a mineralogist (Smith).
by emission spectroscopy (Anderson).
identification (Rice).
by instrumental methods of chemical compounds (Maddin and Rosene).
introduction (Scott).
oxide films on iron in water and oxygen atmospheres
studies by electron microscopy and diffraction (Gulbransen and Copan).
summary (Scott).
industrial
procedure for determination of precision of ASTM Committee D-19 methods. To be published in 1960 Compilation of ASTM Standards on Industrial Water (*see also* Manual on Industrial Water and Industrial Waste Water, *STP 148-D*, p. 203).
Report of Committee D-19, 523.
Symposium on Technical Development in the Handling and Utilization of Water and Industrial Waste Water—Summary of Proceedings of Third Pacific Area Meeting, 50, 51 (*to be published as STP 273*):
conservation and pollution control programs cooling towers (Finnerty).
determination of radioactive materials (Barker).
industrial waste
effects on waters for irrigation use (Wilcox).
radioactive, disposal of at Hanford (Schwendiman, Brown, Honstead, Linderoth, and Pearce).
municipal supply of Coalinga, Calif.
batch-type and continuous electric membrane demineralizers (Katz).
municipal waste treatment (Kinney).

- oil field brines
  - disposal in Central Valley, Calif. (Morris, Jr.)
- saline
  - solar distillation
    - present status and material problems (Lappala, Yaeger, and Bjorksten).
- sea
  - field testing of corrosion inhibitors (Wright).
  - treatment for once-through reactor cooling (Richman).
- *See also* Proposed Method.

Waterproofing
- Report of Committee D-8, 466.

Water-Retardants
- external coatings (Soroka). Published in ASTM Bulletin, No. 240, Sept., 40 (TP 170) (*R*).
- *See also* Admixtures.

Wax
- Report of Committee D-21, 542.

Weight Test
- for cotton fibers
  - causticaire method
    - proposed method. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix XIV), p. 830.

Wet-Abrasion Resistance Test
- for coating films (Diamond). Published in ASTM Bulletin, No. 236, Feb., 48 (TP 58) (*R*).

Whitewares
- Report of Committee C-21, 416.

Window Assemblies
- testing
  - Symposium on Testing Window Assemblies. Summary of Proceedings of *STP 251*, 1337:
    - factory-sealed double glazed units (Wilson, Solvason, and Novak).
    - introduction (Crepps).
    - large glass installations
      - structural testing (Frownfelter).
    - methods
      - need for research and development (Dunn).
  - wind and rain penetration
    - Norwegian testing methods (Svendsen and Wingen).

Wood
- compressive strength at elevated temperatures (Ingberg), 1320.
- in building construction
  - Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 46, 48 (*to be published in STP 275*):
    - laminated beams
      - strength studies of scarf joints (Longworth and Stieda).
    - quality control and inspection (Hanrahan).
    - renaissance of
      - in school building construction in California (Maag).
    - structural design
      - deflection limitations (Eby).
      - need for special engineering education (Glaze).
    - termite control
      - in California residential construction (Ebeling).
    - western
      - screw holding ability: effects of test variables (Johnson).
    - wood-pole
      - strength and related properties (Erickson and Wood).
      - type buildings (Keeney).
- large roof beam
  - testing models (Snodgrass)—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 275*).
- Report of Committee D-7, 460.
- treated for marine use
  - Symposium on Wood Treated for Marine Use—Summary of Proceedings of Third Pacific Area Meeting, 45 (*to be published in STP 275*):
    - analysis of creosote
      - use of column chromatography (Drisko and Hochman).
    - evaluating toxicity of chemical agents to the marine borer Limnoria Tripunctata (Vind and Hochman).
    - preservative retention in piling
      - determination by assay of borings (Baechler).
    - pressure-treated Douglas fir and Southern pine
      - marine exposure tests (Graham and Miller).
    - screening of wood preservatives
      - use of small panels (Roe and Hochman).
    - wood-piles in sea water
      - extending life of (Wakeman and Whiteneck).
      - proposed standard rating for evaluation (Dockweiler and Stover).
- yard lumber of common grade
  - determining useful strength (May)—Symposium on Methods for Testing Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 40 (*to be published in STP 275*).

Wood-Pole
- strength and related properties (Erickson and Wood)—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 275*).
- type building (Keeney)—Symposium on Wood in Building Constructions—Summary of Proceedings of Third Pacific Area Meeting, 48 (*to be published in STP 275*).

Wool
- fabric and yarns
  - fiber fineness
    - proposed method. Published in 1959 Special Compilation of ASTM Stand-

Wool (*continued*)
ards on Textile Materials (appendix XV), p. 835.
moisture content
recommendation for standard. Published in 1959 Special Compilation of ASTM Standards on Textile Materials (appendix IX), 808.
Writing, Technical (Walker). Published in ASTM BULLETIN, No. 240, Sept., 20; (Mowbray). Published in ASTM BULLETIN, No. 241, Oct., 18.

X

X-ray
and gamma rays
environmental measurement by removal dose (Hickmott)—Symposium on Radiation Effects and Dosimetry—Summary of Proceedings of Third Pacific Area Meeting, 47 (*to be published in STP 276*).
Symposium on Spectroscopy (Session II)—Summary of Proceedings of Third Pacific Area Meeting, 41 (*to be published in STP 269*):
application to unsolved problems in geochemistry (Adler).
fluorescence
basic practices (Birks).
instrumentation (Kemp).
spectrography
quantitative light element analysis
Fe to Mg for portland cement (Curley).
X-ray Analysis
quantitative
of portland cement (Brunauer, Copeland, Kantro, Weise, and Schulz), 1091.
X-ray Diffraction
of steels and tool steels (Weiss) (appendix I to Sachs, Sell, and Brown paper), 655.

Y

Yarn
number conversion table. Published in 1959 Compilation of ASTM Standards on Textile Materials (appendix III), p. 784.
Report of Committee D-13, 492.
Yttrium
and alloys
mechanical properties (Carlson, Bare, and Gibson)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 272*).

Z

Zaca-Wigmore Experimental Project
Symposium. Symposium on Road and Paving Materials (Session I)—Summary of Proceedings of Third Pacific Area Meeting, 48, 49 (*to be published in STP 277*):
cooperative test series on asphalts (Skog).
correlation of durability tests with field hardening (Simpson, Miles, and Griffin).
progress report (Hveem, Zube, and Skog).
Zinc
alloys, die casting base
spectrochemical analysis
suggested method
by the point-to-plane spark and a-c arc techniques (*issued as separate reprint*).
corrosiveness of test sites
Report of Subgroup of Subcommittee VII of Committee B-3 (appendix II), 183.
Report of Committee B-2, 171.
Report of Committee B-6, 262.
Zircaloy
analysis of
by coulometric determination of tin with electrically generated iodine (Freund and Caton)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 54 (*to be published in STP 272*).
tensile and corrosion properties
effects of heat treatments (Goodwin, Rubenstein, and Shubert)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 272*).
Zirconium
Report of Committee B-2, 171.
test methods
used in the Naval reactor program (Kall)—Symposium on Newer Metals—Summary of Proceedings of Third Pacific Area Meeting, 53 (*to be published in STP 272*).

1959 LIST OF SPECIAL TECHNICAL PUBLICATIONS

The publications listed below have been compiled during the year 1959 to be issued as separate publications not included in either Proceedings or ASTM Bulletin. For a list of earlier Special Technical Publications, see the original listings in Proceedings, Vol. 48, page 1352, and the supplementary listings in Vol. 49, page 1240, Vol. 50, page 1490, Vol. 51, page 1322, Vol. 52, page 1309, Vol. 53, page 1197, Vol. 54, page 1388, Vol. 55, page 1250, Vol. 56, page 1498, Vol. 57, page 1409, and Vol. 58, page 1420. A complete list of Special Technical Publications covering all publications, up to and including 1950, is included in the 50-Year Index to ASTM Publications. Also a complete list up to and including 1955 is published in the 5-Year Index to ASTM Publications (1951–1955).

Special Technical Publication Number	Title
41-D	Index to the Literature on Spectrochemical Analyses, Part IV, 1951–1955 (1959)
56-M	1958 Supplement to the Bibliography and Abstracts on Electrical Contacts (1959)
125-A	1959 Supplement to the Bibliography of Spectrophotometric Methods of Analysis for Inorganic Ions (1959)
148-D	Manual on Industrial Water-1959 Printing (1959)
251	Symposium on Testing Window Assemblies (1959)
252	Bituminous Paving Materials (1959)
253	Symposium on High Voltage and Cable Insulation
254	Soils—1959 Annual Meeting (1959)
255	Report on Marine Atmosphere Exposure of Galvanic Couples Involving Magnesium (1959)
256	Symposium on Identification of Water-formed Deposits (1959)
257	Symposium on Microscopy (1959)
258	Symposium on New Visual Aids for Standardizing and Communicating Product Appearance (1959)
259	Symposium on Spectroscopic Excitation (1959)
260	Literature Surveys on Influence of Stress Concentrations and Elevated Temperatures and the Effects of Nonsteady Load and Temperature Conditions on the Creep of Metals
261	Symposium on Radioisotopes in Metals Analysis and Testing (1959)
262	Symposium on Electron Metallography (1959)
263	Symposium on Education in Materials (1959)
264	Report on Stress-Corrosion Cracking of Austenitic Chromium - Nickel Stainless Steels (1959)
265	Symposium on Electroless Nickel Plating (1959)

Erratum.—Omitted in 1957 listing of Special Technical Publications:

148-C Manual on Industrial Water—1957 Printing (1957)